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BULLETIN No. 51.

U. S. DEPARTMENT OF AGRICULTURE.

DIVISION OF CHEMISTRY.

PROCEEDINGS

OF THE

FOURTEENTH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS

HELD AT

WASHINGTON, D. C.,

OCTOBER 26, 27, AND 28, 1897.

EDITED BY

HARVEY W. WILEY,

SECRETARY OF THE ASSOCIATION.

WASHINGTON:

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LETTER OF TRANSMITTAL.

U. S. Department of Agriculture,
Division of Chemistry,
Washington, D. C., January 17, 1898.

Sir: I beg to transmit herewith, for your inspection and approval, the manuscript of the proceedings of the fourteenth annual meeting of the Association of Official Agricultural Chemists, and ask that it be printed as Bulletin No. 51 of the Division of Chemistry.

H. W. Wiley,
Chief of the Division of Chemistry and Secretary of the Association of Official Agricultural Chemists.

Hon. James Wilson,
Secretary of Agriculture.

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PROCEEDINGS OF THE FOURTEENTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

FIRST DAY.

TUESDAY—MORNING SESSION.

The fourteenth annual convention of the Association of Official Agricultural Chemists was held in the lecture hall of the Columbian University, beginning October 26, 1897. The meeting was called to order by the president, Mr. William Frear, who appointed Messrs. Patterson, Davidson, and Hills a committee on order of business. The committee reported as follows:

First. Report on nitrogen.
Second. Report on potash.
Third. Report on phosphoric acid.
Fourth. Report on soils and ash.
Fifth. Report on foods and feed stuffs.
Sixth. Report on food adulteration.
Seventh. Report on dairy products.
Eighth. Report on sugars.
Ninth. Report on fermented and distilled liquors.
Tenth. Report on tannin.

The election of officers was made a special order for Wednesday, October 27, at 3 p. m.

Upon motion the report was adopted.

The president read the following annual address to the association.

ADDRESS OF THE PRESIDENT.

MEMBERS OF THE ASSOCIATION OF OFFICIAL CHEMISTS: It is customary, at the opening of each annual convention of the association, for your presiding officer to make a few introductory remarks relative to its aims and work. It is well for the ship if the officers and the lookouts be not satisfied with the progress indicated by the beating throb of the engine, but are also keenly observant of her course and her speed, keeping a watchful eye upon compass and log.

It is likewise well for such an association as ours if those concerned in its work are not content simply with the performance of much routine

work along lines already well fixed by the earlier work of the association, but study also the logic of its progress and growth, the new demands each step in the science of the chemist, the art of the technologist, and the statute of the legislator makes upon it.

The early inception of the idea of this organization was due to the friction between buyer and seller and their arbitrators, the chemists, resulting from differences in the methods of determining the phosphoric acid in that ill-defined group of materials called "reverted phosphates". The purpose of the association was, by study of details of method, to arrive at uniformity of analytical processes; and, by comparison of results of manipulation in the laboratories concerned, to arrive at identity of analytical results by different chemists upon the same material.

The striking benefits that followed these efforts induced those interested in the work of the agricultural experiment stations, organized not long after, to attempt a similar unification of methods for the determination of the ill-defined groups of substances whose separation is attempted in the course of investigations upon animal nutrition. Work upon soil and ash analysis, upon dairy products, and upon other products of rural industry, such as sugar products, wines, etc., was undertaken soon after.

Finally, the growing recognition of the value of chemical analysis in the control of tanning operations has led to the formation of a group of workers upon the methods applicable to the analysis of tanners' raw materials and their products.

Primarily, the work of the association has been chiefly along the lines of importance to the official chemist. This must still be, to a large extent, true of the association's work. But it will fail of its high opportunities and choose an ideal lower than it may properly select if its work be not pushed also, in a large measure, along more distinctly scientific lines.

The work in determination of available plant food in soils, the study of the methods of analysis applicable to slag phosphates, the test of various methods for the separation and determination of simple substances and narrower, better-defined groups of substances in cattle foods and dairy products, the studies of solubilities of various phosphates, may be cited as illustrations of valuable work by the association in this field. If we would accomplish most, we must remember that the ideal accomplishment is not the perfection of an arbitrary method for the determination of an ill-defined group of substances, but, much further on, the attainment of a method by which such a group may be resolved into its simple components, and the effective value of each determined. For example, the work upon phosphoric acid will not be finished by the shaping of a method capable of yielding excellent duplicate results upon dissolved phosphate, raw phosphates—calcic and ferruginous—upon bones and slag alike; but only when, by the methods devised (probably a separate method for each), it is made possible to gain an idea of the available plant food that each will yield under some

normal condition, or, it may be, under several distinct classes of conditions. This is, indeed, a far more difficult field of operation, but it is none the less important.

It may be well, at this point, to refer to an impression prevailing in the minds of some of our associates. As an incident to the perfection of fertilizer methods there came the opportunity of comparison of the results of manipulation by different analysts. A valuable training was thus afforded to the young beginners with whom our laboratories, sprung suddenly into existence, were largely manned. The opportunity thus afforded is extremely valuable in its influence both upon the accuracy of the analyst and upon the confidence of the man whose goods he values. Some, indeed, came to regard this as the chief *raison d'être* of the association, and have suggested the desirability of so conducting its work that it shall become practically a "moot court" for tyros in analysis. Without depreciating at all the value of such comparison, I protest earnestly against such a conception of the work to be done. There is ample scope for the exercise of generalship on the part of our more experienced members, as well as for drill to the juniors of our organization.

With these brief comments upon the general work of the association, I desire to again call your attention to the fact that the prime work of the association is along lines important to the official agricultural chemist. At first his work was confined almost exclusively, in America, to the fertilizer control. The association's transactions in this domain have been of great helpfulness to those concerned in the analysis of fertilizers, whether as manufacturers, as sellers, as buyers, or as control officials.

But parallel with the fertilizer control work, there has arisen a demand upon the agricultural chemist for the application of analytical methods to the detection of food adulterations, with a view to insuring their prevention. For the chemist engaged in this work the association has done little that is directly and specifically useful—this is said with full remembrance of our work on milk and its products, sugar products, and alcoholic liquors. Our effort has been chiefly directed toward the perfection of methods devised to determine the normal constituents of certain food materials, scarcely at all to the detection and estimation of foreign admixtures. The former work should be continued, but there is a pressing demand for the latter. I wish particularly to note a few facts and to offer a few suggestions with reference to this latter work.

It may not be amiss to consider the great values concerned. Waldron (Handbook of Currency and Wealth, 1896, p. 101) estimates the nation's expenditure for food to be $3,305,000,000, and for liquors $900,000,000, making a grand total of $4,205,000,000, or nearly 31 per cent of the entire expenditure for necessaries, luxuries, and capital; and this grand total does not include those articles of food properly classed as luxuries.

Edge (Rept. Pa. Dept. of Agriculture, 1895, p. 215) estimates the value of the food consumed in the country at $4,500,000,000; and Commissioner McNeal, of Ohio, that of foods, drinks, and drugs at $7,280,000,000.

From the records prepared under the direction of Hon. Carroll D. Wright, United States Commissioner of Labor (Sixth and Seventh Annual Reports), showing the earnings and itemized expenditures of 6,809 families, containing 35,312 persons, representing the iron and steel workers, bituminous coal miners, ore miners, limestone quarrymen, glass workers, and cotton and wool workers of the country, I find that the average family spent upon its food 39.4 per cent of its total income—a proportion much larger than that given by Waldron, who fixed the average at 24.23 per cent. These figures from Wright's records do not include expenditures for alcoholic liquors. If applied to the whole population, they would bring the total to $3,244,000,000; of this amount 52 per cent, or $1,687,000,000, was spent upon adulterable foods.

The following statement of the values of the food products manufactured in the United States in 1889 I have summarized from the figures of the Eleventh Census:

Flour, milling and bakery products (less the materials used in bakeries)	$569,885,430
Fruit, vegetables, food preparations, pickles (including canned oysters)	63,991,790
Sugars, sirups (refining), glucose, and confections (less materials used by confectioners)	155,755,417
Dairy products, lard, and oleo	71,149,146
Condiments, flavoring extracts, salt, and vinegar (including cider)	22,155,238
Table beverages and bottled drinks (including spices)	92,847,103
Alcoholic liquors	289,775,639
Total	1,265,559,763

But this category omits many highly important food products—not only such as undergo no manufacturing process before they come to the kitchen and are little liable to adulteration, such as fresh meats, eggs, fruits, vegetables, and nuts—but also canned and pickled meats and fish, honey, crude sirups and sugars, and milk. Neither does it include those materials ready for family use.

The census figures show the following amounts for certain of these products in 1890:

Products.	Amount.	Price.	Total.
		Cents.	
Honey ..... pounds..	63,897,327	5	$3,194,866.35
Cane molasses ..... gallons..	25,409,228	30	7,622,768.40
Sorghum molasses ..... do....	24,235,219	28	6,785,861.32
Maple sirup ..... do....	2,258,376	60	1,355,025.60
Maple sugar ..... pounds..	32,952,927	8	2,636,234.16
Roughly valued at	.....	.....	21,594,755.83

The Eleventh Census makes the total value of the homemade and factory-made dairy products and of the milk not transformed into manufactured products $411,976,522. Alvord (Bulletin No. 11, Bureau of Animal Industry) makes a higher estimate, $454,900,000, which, in view of the fact that the census figure includes statements from only such factories as made returns, and many failed to make the proper returns, is probably not too high. Alvord includes the yield of town cows, omitted by the census.

Much, however, of this dairy product never leaves the farm. How much is sold it is difficult to estimate. From my own observation I incline to believe that the per capita consumption of whole milk, butter, and cheese is less on the farm than elsewhere.

In 1890 the per capita production of butter, according to the census, was 19 pounds, deducting the amount exported; 21.7 pounds according to Alvord's estimates; of cheese, according to the census, 3.7 pounds; according to Alvord, 4.1 pounds. Alvord also estimates the average direct milk consumption at 25½ gallons of whole milk per capita.

In 1890 Boston had a population of 488,477; it has probably grown somewhat since then. Correspondence with Hon. George M. Whitaker, executive officer of the Massachusetts dairy bureau, places the annual milk delivery in Boston at about 31,800,000 gallons; and of butter—based upon five months' returns, less corresponding exports—at 54,067,200 pounds. Estimating that the market supplies 600,000 population with butter and 500,000 with milk, this makes the annual consumption per capita 90 pounds of butter and 60⅓ gallons of milk. The amount of both butter and milk made up into other food preparations and exported in such guise I have not been able to estimate.

In 1890, New York had a population of 1,515,301. The New York dairy and food commissioner states the annual consumption of that city to be:

Butter	pounds	85,880,442
Cheese	do	38,224,752
Condensed milk, cream, and whole milk, equivalent to the whole milk (*circa*)	gallons	107,410,000

Equivalent per capita, assuming a present population of 1,750,000, to—

Butter	pounds	49.1
Cheese	do	21.8
Milk	gallons	61.4

These per capita figures, while doubtless excessive,[1] indicate, nevertheless, that the rate of consumption of these dairy products on the

[1] In a recent letter Mr. Whitaker says: "The city of Boston proper is surrounded by a circle of towns and cities. The business interests of these places are united with the business interests of Boston, and they are collectively called 'Greater Boston.'

"The milk of Greater Boston comes, first, by rail from a territory roughly stated as between 25 and 150 miles distant from Boston; second, by wagon from a territory

farm must be far less than the most urban communities. So that an assignment of one-fourth of these dairy products to the rural communities, a proportion equal to that their working population bears to the entire working population of the country, leaves a balance for dairy products sold upon the market and liable to adulteration that is certainly too low. This balance, after deducting the nearly $63,000,000 worth of dairy products made in the factories, is about $350,000,000 worth.

For pickled and canned meats I have obtained no satisfactory figures.

So many of our imports of food materials have already been accounted for as raw materials, that I will not consume your time by any elaborate showing of their importance, but will simply note the following items for the year ending June 30, 1896:

Bottled drinks, nonalcoholic	$288,963
Macaroni	599,836
Infants' foods	66,545
Lemon juice, etc	84,073
Vanilla bean	1,013,609
Chocolate, chicory, cocoa, and coffee, with substitutes	88,151,593
Farinaceous substances	257,588
Anchovies	956,255
Malt liquors	1,652,971
Vegetable oils	2,688,051
Extract of beef	214,932
Rice flour	916,512
Saccharin	53,760
Salt	752,108
Spices	2,137,197
Mustard	227,928
Distilled spirits	2,098,122
Sugar, molasses, and confectionery	84,369,504
Tea	12,688,740
Vegetables, canned and pickled	1,045,999
Waters, soda, etc	585,694
Wines	7,212,997

When it is further shown, as the records of our various State food controls amply prove, that from 5 to 15 per cent of the entire food

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within 25 miles of the city; and third, a small amount is produced in this territory, for as we go out of Boston some of the towns shade off to agricultural communities and keep quite a number of cows. The figures which I gave you refer to the milk supply of Greater Boston.

"Second, as to your inquiry about butter: The expression 'butter exported' means butter exported across the water, particularly to England. The Chamber of Commerce receives from the railroads the amount of butter brought to the city. From this the butter exported is deducted, and the balance is called 'domestic consumption.' This is butter which goes into the ordinary lines of trade from Boston as a commercial center, but is almost entirely consumed in eastern Massachusetts, southern New Hampshire, and a little in Rhode Island. Occasionally a drummer finds his way into the western part of the State, but that is an agricultural community and very largely supports itself. The same is true for eastern Connecticut. Western Connecticut is principally supplied from the New York market, and New York firms compete with Boston firms for the business of Rhode Island."

supply upon the markets is adulterated, and that fully 10 per cent of the adulteration is injurious to health, little further is needed, it seems to me, to emphasize the importance of the work of the food control and of all subjects relating thereto.

There are two ways in which this association can aid the food-control chemist:

(1) By the careful selection, accurate description, and test of methods fitted for the control examination of the various classes of adulterable food materials upon the market. Food-control chemists differ to-day in their results even more than fertilizer-control chemists differed when this association was organized, and both from the same cause, namely, the use of different methods for the same determination. The prevalence of these differences among reputed experts seriously impairs the confidence of judicial and administrative officers in the chemist's work and begets a justifiable timidity on the part of the honest manufacturer and dealer, whose hearty cooperation is most important to the success of the control. All concerned in this work have much to hope for from the unification and improvement of analytical methods in this field of operation. Toward this end the association looked in the appointment last year, for the first time, of a reporter on food adulteration.

(2) By taking steps to secure the establishment of standards of composition for pure food substances, just as the druggists have done for drugs. The United States Pharmacopœia is the accepted authority as to standards for the purity of drugs, and incidentally for a few food substances. Most State and municipal laws relating to the control of drug adulteration distinctly accept the pharmacopœial standards of purity. A similar work must be done for foods. In the absence of such an accepted set of standards each food chemist is compelled to formulate his own. The result is that the standards used by different chemists are based upon insufficient data and are formulated according to different canons, with the consequence that legal actions are instituted in good faith by control officers only to be lost by the failure of the several experts to agree upon the essential bases of comparison.

Another result of this lack of consensus of expert opinion, based upon the most reliably ascertained facts of food composition, is the incorporation into legislation of arbitrary standards that work serious injustice to innocent producers and dealers.

I earnestly hope that this year this association may, from its vantage point of successful attainment in other directions, begin a movement to overcome these difficulties under which the official food analyst to-day labors.

MEMBERS AND VISITORS PRESENT.

The following members and visitors were present:

Allen, E. W., Office of Experiment Stations, United States Department of Agriculture, Washington, D. C.
Anderson, James T., Alabama Experiment Station, Auburn, Ala.
Atwater, B. M., Syracuse, N. Y.

Bartlett, J. M., Agricultural Experiment Station, Orono, Me.
Beal, W. H., United States Department of Agriculture, Washington, D. C.
Bigelow, W. D., Division of Chemistry, United States Department of Agriculture, Washington, D. C.
Bryant, A. P., Storrs Experiment Station, Middletown, Conn.
Carpenter, F. B., Virginia-Carolina Chemical Company, Richmond, Va.
Carr, Oma, Corry, Pa.
Chesnut, V. K., Department of Agriculture, Washington, D. C.
Clark, Reed Paige, Washington, D. C.
Coates, Charles B., jr., Louisiana State University, Baton Rouge, La.
Craighill, G. P., Lynchburg, Va.
Crampton, C. A., Treasury Department, Washington, D. C.
Cutter, W. P., United States Department of Agriculture, Washington, D. C.
Davidson, R. J., Experiment Station, Blacksburg, Va.
Dickeson, William E., United States Appraiser's Office, Philadelphia, Pa.
Ellett, W. B., Experiment Station, Blacksburg, Va.
Ewell, E. E., Department of Agriculture, Washington, D. C.
Fernandez-Cavada, Fernando, Audubon Park, New Orleans, La.
Frear, William, Experiment Station, State College, Pa.
Fulmer, Elton, Experiment Station, Pullman, Wash.
Gaines, Richard H., Department of Agriculture, Richmond, Va.
Hand, W. F., Agricultural College, Miss.
Haywood, J. K., United States Department of Agriculture, Washington, D. C.
Hills, Joseph L., Experiment Station, Burlington, Vt.
Hird, J. D., Washington, D. C.
Hite, B. H., Experiment Station, Morgantown, W. Va.
Huston, H. A., Experiment Station, Lafayette, Ind.
Jenter, C. G., Experiment Station, Geneva, N. Y.
Jones, Charles H., Experiment Station, Burlington, Vt.
Kilgore, B. W., Agricultural College, Miss.
Knox, Roy, Morgantown, W. Va.
Krug, William H., United States Department of Agriculture, Washington, D. C.
Krumbhaar, Charles Conrad, Audubon Park, New Orleans, La.
Langworthy, C. F., United States Department of Agriculture, Washington, D. C.
Laughlin, J. R., Maryland Agricultural College, College Park, Md.
Lehmann, G. W., Chemist, Board of Health, Baltimore, Md.
Lindsey, J. B., Hatch Experiment Station, Amherst, Mass.
McDonnell, H. B., Maryland Agricultural College, College Park, Md.
Moore, C. C., United States Department of Agriculture, Washington, D. C.
Munroe, C. E., Columbian University, Washington, D. C.
North, J., 205 Third avenue, New York City, N. Y.
Patrick, George E., United States Department of Agriculture, Washington, D. C.
Patterson, H. J., Experiment Station, College Park, Md.
Peacock, S., Philadelphia, Pa.
Penny, C. L., Experiment Station, Newark, Del.
Perkins, George E., 72 Jackson street, Providence, R. I.
Persons, A. A., Experiment Station, Lake City, Fla.
Ross, B. B., Auburn, Ala.
Runyan, E. G., United States Department of Agriculture, Washington, D. C.
Schweinitz, de, E. A., Department of Agriculture, Washington, D. C.
Scovell, M. A., Experiment Station, Lexington, Ky.
Skinner, W., Maryland Agricultural College, College Park, Md.
Spencer, G. L., United States Department of Agriculture, Washington, D. C.
Street, John Phillips, Experiment Station, New Brunswick, N. J.
Sweetser, W. S., Experiment Station, State College, Pa.

Thigpen, J. H., Washington, D. C.
Trescot, T. C., United States Department of Agriculture, Washington, D. C.
Van Slyke, L. L., Experiment Station, Geneva, N. Y.
Veitch, F. P., Maryland Agricultural College, College Park, Md.
Wainwright, J. H., United States Appraiser's Office, New York City, N. Y.
Welty, Harry T., College Park, Md.
Wheeler, Edward J., Department of Agriculture, Albany, N. Y.
Wheeler, H. J., Experiment Station, Kingston, R. I.
Wiley, H. W., United States Department of Agriculture, Washington, D. C.
Williams, C. B., Experiment Station, Raleigh, N. C.
Winton, A. L., Experiment Station, New Haven, Conn.
Withers, W. A., Experiment Station, Raleigh, N. C.
Yocum, J. H., United States Leather Company, New York City, N. Y.

The PRESIDENT. It is customary at this time to appoint a committee to acquaint the honorable Secretary and Assistant Secretary of Agriculture with the fact that we are in session and request their presence at such time as they may find it possible to come. If there be no objection, the Chair will now appoint as such committee the vice-president of the association, Mr. Winton, and Mr. Kilgore, of Mississippi.

Upon Mr. Wiley's suggestion, the committee was directed to invite the Secretary (the Assistant Secretary being out of town) to be present on Wednesday morning at 11 o'clock, at which hour probably a larger number of members would be present.

Mr. Street, of the New Jersey station, then read the report on nitrogen, as follows:

REPORT ON NITROGEN.

By JOHN PHILLIPS STREET, *Referee.*

The work for the year will be considered under three heads.

I. The determination of nitric nitrogen.

II. The determination of total nitrogen.

III. The determination of the availability of organic nitrogen.

Early in January a letter requesting cooperation was addressed to fifty-five chemists, representing all of the American experiment stations and several commercial firms. Twenty-five favorable replies were received to this letter, fifteen chemists agreeing to do the regular work and ten the work on organic nitrogen. Of this number, eight have reported results on nitric and total nitrogen and seven on organic nitrogen. On February 8 a letter of instruction was sent to those who had signified a wish to take part in the work.

PREPARATION OF SAMPLES.

Three samples of fertilizers were prepared for distribution. The materials used in compounding these mixtures were nitrate of soda, cotton-seed meal, acid phosphate, potassium chlorid, and Lobos guano. The sample of nitrate of soda was prepared by recrystallizing a quantity of commercial nitrate twice, with a final evaporation to dryness. No attempt was made to secure an absolutely pure product, and traces of chlorids, sulphates, and moisture were shown to be present by subsequent tests. The Lobos guano was a complete fertilizer, the source of whose nitrogen was a natural guano. It was thought by the referee that the use of such a material would be as severe a test of the methods as could be desired.

These ingredients were dried for five hours at from 60° to 70° C., and then were allowed to stand exposed to the air for fifteen hours, in order that they might become in equilibrium with the atmosphere. They were then ground to pass through a one twenty-fifth inch sieve, except the nitrate of soda and potassium hlorid, which were passed through a one fiftieth-inch sieve. The samples were then thoroughly mixed and portions selected for analysis, the results of which follow:

Composition of samples.

Substance.	Nitric nitrogen.	Ammonia.	Organic nitrogen.	Total nitrogen.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Nitrate of soda	16.25	16.25	.....	16.25
Cotton-seed meal	.....	.....	6.93	6.93
Lobos guano	.33	1.99	.94	3.26

No traces of nitrogen were found in the acid phosphate or potassium chlorid.

The ingredients of the different mixtures were then spread out on a heavy paper and thoroughly mixed by turning with a spatula. They were then bottled and securely sealed.

The mixtures were made up as follows:

Sample 1:
1,000 grams nitrate of soda, containing 162.50 grams nitrogen,
Or 16.25 per cent nitric nitrogen.

Sample 2:
100 grams nitrate of soda. containing 16.25 grams nitrogen;
300 grams cotton-seed meal, containing 20.80 grams nitrogen;
500 grams acid phosphate;
100 grams potassium chlorid,
Or 3.71 per cent total nitrogen, containing 1.63 per cent nitric nitrogen.

Sample 3:
100 grams nitrate of soda, containing 16.25 grams nitrogen;
900 grams Lobos guano, containing 29.34 grams nitrogen,
Or 4.56 per cent total nitrogen, containing 1.66 per cent nitric nitrogen and 1.79 per cent as ammonia.

I.—The Determination of Nitric Nitrogen.

The work on nitric nitrogen consisted in a test of the Ulsch-Street method in comparison with the Scultze-Tiemann method. It was the expressed sentiment of the last convention that the Ulsch-Street method should be given a fair and general trial this year. In light of this fact, it was hoped that a large number of analysts would test it in their work. The chemists who objected so strongly to the adoption of this method last year have shown their deep interest in its success this year by refraining, with hardly an exception, from using it in a single test.

The modified Ulsch method recommended was the same as that given in Bulletin 49, United States Department of Agriculture (p. 13), except that from 7 to 10 grams of heavy oxid of magnesia free from carbonates were suggested instead of the smaller quantity previously used. The Schulze-Tiemann method used was that given by Fresenius in his Quantitative Analysis (Amer. Ed., 1886, pp. 473–475).

Comparison of methods.

Analysts.	Nitrate of soda.								
	No. 1.					No. 2.		No. 3.	
	Ulsch-Street.	Schulze-Tiemann.	Kjeldahl.	Gunning.	Absolute.	Ulsch-Street.	Schulze-Tiemann.	Ulsch-Street.[1]	Schulze-Tiemann.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
T. C. Trescot, U. S. Dept. of Agr.	16.23	16.22	.....	.....	16.26	1.64	1.60	1.77	1.72
H. K. Miller, N. C. Station	16.30	16.28	.....	.....	.....	1.69	.....	1.74	.....
C. H. Jones, Vt. Station	[2]15.99	.....	.....	.....	.....	1.61	.....	1.76	.....
F. B. Bomberger, Md. Station	16.14	.....	.....	16.10	.....	1.63	1.64	1.68	1.69
R. J. Davidson, Va. Station	16.13	.....	.....	.....	.....	1.70	.....	1.72	.....
C. S. Cathcart, Lister's Agricultural Chemical Works	16.12	.....	.....	.....	.....	1.70	.....	1.61	.....
A. M. Peter, Ky. Station	[2]15.70	.....	.....	.....	.....	1.71	.....	1.68	.....
H. E. Curtis, Ky. Station	[2]15.66	.....	16.25	16.14	.....	1.70	.....	[2]1.51	.....
J. P. Street, N. J. Station	16.24	16.32	.....	.....	.....	1.71	1.62	1.73	1.66
A. W. Ogden, Conn. Station	16.20	.....	.....	.....	.....	1.70	1.52	1.77	1.62
Average	16.19	16.27	16.25	16.12	16.26	1.68	1.60	1.72	1.67
Theory	16.25	.....	.....	.....	.....	1.63	.....	1.66	.....

[1] 1.86 per cent ammonia deducted.

[2] Omitted from the averages.

COMMENTS OF ANALYSTS.

T. C. Trescot.—The results obtained on sample 1 by the Ulsch-Street method were very unsatisfactory. The result reported is the average of the three highest (16.21, 16.22, and 16.28) of many determinations.

H. K. Miller.—In working on sample 1 by the Ulsch-Street method I found it necessary to use dropping bulbs to prevent loss. I was unable to get satisfactory results with the Schulze-Tiemann method, as I was unable to get apparatus perfectly tight; and in working with samples 2 and 3 by this method frothing was so bad that I had to give up making determinations on these two samples. I used caustic soda, also, in determining nitric nitrogen by the Ulsch-Street method in Nos. 2 and 3 and obtained higher results, though I send you only the results obtained from using magnesium oxid.

A. M. Peter.—Instead of using the small amount of water, as per your instructions, and distilling nearly to dryness, I used 300 cc of water, and in the case of Nos. 2 and 3 mixed the magnesium oxid with part of this water to a cream, for the sake of more thorough action, and distilled off about 220 or 230 cc. By subsequently carrying the distillation nearly to dryness, it was found that in each case practically all the ammonia had been obtained in the first distillate. The largest amount obtained in the second distillation was only 0.07 per cent in No. 2, the others being mere traces or none at all. My idea is that if from 300 to 350 cc of water be used and the precaution taken to mix the magnesium oxid with part of the waters so that it will not form lumps and interfere with the action, there will be no difficulty in obtaining all the ammonia by distilling off not less than two-thirds of the total volume of the liquid.

A. W. Ogden.—We found the Ulsch-Street method as applied to nitrate salts very satisfactory indeed. We will probably use it next year in the analysis of our nitrate of soda samples. The Ulsch-Street method gave very good results on mixed fertil-

izers, although it is not certain that it will prove in our hands, all things considered, superior to our old standby, the Schulze-Tiemann.

Ten chemists reported results by the modified Ulsch method. The results this year on the nitrate of soda sample are a decided improvement on those of past years. Seven of the ten analysts secure results within 0.13 per cent, while four are within 0.05 per cent of theory. One of the others is 0.26 per cent low, while the other two, representing the work of two chemists at the same station, are from 0.55 to 0.59 per cent low. Eliminating these two results, which were obtained by a method not strictly in accord with that sent out, the difference between the lowest result of the others and theory represents a variation of but 0.016 per cent of the total nitrogen in the sample. Had these results been secured on a 4 per cent sample this variation would represent a difference of but 0.06 per cent.

The results secured on samples 2 and 3 are also excellent, the extreme range between highest and lowest being 0.10 per cent in No. 2, and omitting one result, 0.16 per cent in No. 3. The greatest variation from theory in No. 2 is 0.08 per cent and in No. 3, with one exception, 0.10 per cent. Attention is called to the fact that the low result on No. 3 is reported by the same chemist who reported a low result on No. 1. This would seem to indicate that the method was not altogether responsible for the discrepancies.

The comparison of results by the Ulsch-Street and the Schulze-Tiemann methods, while representing the work of but four chemists, is very satisfactory, the average difference being 0.08 per cent in No. 1, 0.08 per cent in No. 2, and 0.05 per cent in No. 3. It would seem that a fairer comparison of these two methods would be secured by using the results of only those chemists who tried both methods. On this basis we have—

Ulsch-Street.			Schulze-Tiemann.		
No. 1.	No. 2.	No. 3.	No. 1.	No. 2.	No. 3.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
16.23	1.64	1.77	16.22	1.60	1.72
16.30	1.63	1.68	16.28	1.64	1.69
16.24	1.71	1.77	16.32	1.52	1.62
[1]16.26	[1]1.67	[1]1.74	[1]16.27	[1]1.60	[1]1.67

[1] Average.

These results are excellent, and show that the Ulsch-Street method, instead of giving low results as has been claimed, has, on the other hand, a tendency to give slightly higher results, the differences being 0.07 per cent in favor of the Ulsch method in both samples 2 and 3. This is quite in accord with the well-known fact that the Schulze-Tiemann gives low results unless the utmost care is exercised in its manipulation.

In a comparison of these methods on samples of commercial nitrate of soda, the referee secured the following results: In a total of thirteen samples the Ulsch method gave higher results in nine samples and lower in four. The greatest variation was 0.11 per cent; the averages were 15.87 per cent, being identical by the two methods.

The results secured by the Ulsch-Street method this year confirm the referee's belief in its accuracy, its efficiency, and its adaptability to the work of this association. That the method requires care in its manipulation is not peculiar to this method alone; and should be no objection to its use. Aside from its accuracy, its rapidity is one of its strongest recommendations. This advantage may not appeal strongly to many of the chemists who have little work to do along this line, but to those stations where several hundred nitrate determinations are required each year, as in the New Jersey Station, a rapid method is essential. As has been pointed out before, nearly one-half the time can be saved by using this method.

II.—The Determination of Total Nitrogen.

At the last meeting of this association the referee was requested to ascertain whether the method called the Fassbänder method was not erroneously named. In obedience to this request, the referee wrote to Dr. Fassbänder, who first called it to the attention of this association, and received the following reply:

Kempen am Rhein, *April 21, 1897.*

Mr. John P. Street, *New Brunswick, N. J.*

Dear Sir: The method which I employed in 1894 in the determination of nitrogen in the presence of nitrates was not first suggested by me, but by Dr. V. Schencke in No. 54 of the Chemiker Zeitung for 1893. It is known here as the Ulsch-Kjeldahl method, and is rapidly coming into general use.

Respectfully, yours,

Dr. Fassbänder.

The work on total nitrogen this year consisted of an examination of the Ulsch-Kjeldahl and Ulsch-Gunning methods in comparison with those already in use. The Ulsch-Kjeldahl and Ulsch-Gunning methods are simply a combination of the Ulsch method with the Kjeldahl and Gunning methods, respectively. The former is the method suggested by Dr. Schencke; the latter was suggested to the referee by Mr. A. L. Winton, of the Connecticut Station, in private correspondence. The methods as outlined by the referee in his letter of instructions are as follows:

ULSCH-KJELDAHL METHOD.

Weigh 1 gram of material into a Kjeldahl flask, cover with 20 cc of water, shake and allow to stand for ten minutes. Then add two grams of powdered iron (reduced iron answers very well) and 10 cc of sulphuric acid (sp. gr. 1.35). Warm gently on furnace until evolution of hydrogen ceases. Add 0.7 gram of mercuric oxid and 20 cc of strong sulphuric acid, and then proceed as in the regular Kjeldahl method.

ULSCH-GUNNING METHOD.

Make the preliminary conversion as above with reduced iron and dilute sulphuric acid. Proceed as in the regular Gunning method as given in Bulletin 46, United States Department of Agriculture, page 17. In samples rich in carbon, as sample No. 2, it is necessary to boil for some time after the liquid becomes colorless.

Comparison of methods.

Analysts.	No. 2.				No. 3.			
	Kjeldahl.	Ulsch-Kjeldahl.	Gunning.	Ulsch-Gunning.	Kjeldahl.	Ulsch-Kjeldahl.	Gunning.	Ulsch-Gunning.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
T. C. Trescot, U. S. Dept. of Agr.	3.73	.....	3.75	3.70	4.51	.....	4.54	4.52
H. K. Miller, N. C. Station	3.83	3.78	.....	3.81	4.67	4.68	.....	[1]4.72
C. H. Jones, Vt. Station	3.62	3.65	.....	3.63	4.50	4.52	.....	4.52
F. B. Bomberger, Md. Station	3.72	3.68	3.76	3.76	4.58	[1]4.83	4.58	[1]4.78
R. J. Davidson, Va. Station	3.74	3.74	.....	3.75	4.61	4.46	.....	4.49
A. W. Ogden, Conn. Station	3.58	.....	3.67	3.66	4.41	4.50	.....	4.42
C. S. Cathcart, Lister's Agricultural Chemical Works	3.72	3.76	3.74	.....	4.58	4.52	4.59	.....
H. E. Curtis, Ky. Station	3.79	3.83	3.77	3.78	4.48	[1]4.28	4.55	4.46
A. L. Emery, Ohlandt Fertilizer Works	3.83	3.82	3.79	3.84	4.65	4.63	4.50	4.66
J. P. Street, N. J. Station	3.68	3.72	.....	3.69	4.49	4.52	.....	4.50
Average	3.72	3.75	3.75	3.74	4.55	4.55	4.54	4.51
Theory	3.71	.....	.....	.....	4.57	.....	.....	.....

[1] Not included in average.

COMMENTS OF ANALYSTS.

T. C. Trescot.—The data obtained by the Ulsch-Kjeldahl method were so unsatisfactory that no report is made of them.

A. L. Emery.—I am unable to understand why the Gunning method on sample 3 should run low. In other comparative results I have found it to agree very closely. I am very much pleased with the Ulsch-Gunning method. It is rapid and the reaction seems to be clean and complete in both low and high grade material.

A. W. Ogden.—The Gunning methods gave rather higher results on sample 2 than the Jodlbauer. These high results I believe are nearest correct and give us a point in favor of the sulphate of potash methods. In our regular fertilizers we have not found such a difference, and we conclude the sample must be extraordinary.

While there are no wide variations, the results by the Kjeldahl method, which is an official method of this association, are not entirely satisfactory. There is a variation of over 0.10 per cent in three results on No. 2 and in two on No. 3, which is greater than we usually expect by this method. The Gunning results are excellent, the greatest variation in No. 2 being 0.08 per cent and on No. 3, 0.06 per cent.

The results secured by both of the new methods are quite satisfactory. With the Ulsch-Kjeldahl method the greatest variation from theory on No. 2 is 0.12 per cent and on No. 3, 0.28 per cent. Six of the eight results on No. 2 vary less than 0.08 per cent and six of the nine results on No. 3 vary less than 0.10 per cent. With the Ulsch-Gunning method the greatest variation from theory on No. 2 is 0.13 per cent and on No. 3, 0.22 per cent. Two results vary by more than 0.10 per cent on No. 2 and three on No. 3. By eliminating the few wide results, we secure averages by the two methods almost identical on both samples. The results obtained by both of these methods are quite as good as those secured by the modified Kjeldahl method in both samples.

One hundred and ninety-four comparative tests have been made between the Ulsch-Kjeldahl and the official methods by Mr. L. H. Merrill, of the Maine Station, and the referee during the last two years. The averages secured by the two methods are identical by both methods. It is believed, however, that this method, as well as the Ulsch-Gunning method, may be further perfected, and the referee does not feel justified in recommending their adoption until a more general trial has been made.

III.—The Determination of the Availability of Organic Nitrogen.

Ten chemists signified a desire to receive samples for the study of the availability of organic nitrogen; of this number, seven have returned results.

It was the plan of the referee to make a further study of the pepsin-hydrochloric acid and the permanganate of potash methods. For this purpose ten samples of organic nitrogenous materials were prepared. These samples were carefully dried, ground, bottled and sealed as described in the earlier part of this report. A description of the samples follows:

No. 4. Dried blood.—A high-grade sample in good mechanical condition.

No. 5. Cotton-seed meal.—A fine yellow sample of the decorticated meal, containing 3.74 per cent phosphoric acid and 1.99 per cent potash.

No. 6. Dried ground fish.—Containing 8.02 per cent phosphoric acid.

No. 7. Tankage.—An average sample, containing 14.78 per cent phosphoric acid.

No. 8. Hoof meal, "E" brand.—A fine dry product.

No. 9. Raw leather.—Pure sole-leather shavings obtained from a local factory.

No. 10. Wool waste.—An unusually good sample of this product, containing 0.53 per cent phosphoric acid and 2.42 per cent potash.

No. 11. Bone sawings.—A hard, dry bone obtained from a button factory. It contained 25.42 per cent phosphoric acid.

No. 12. Steamed bone.—In very fine mechanical condition, and containing 26.92 per cent phosphoric acid.

No. 13. Raw bone meal.—An average sample, containing 22.70 per cent phosphoric acid.

The value of work of this kind is greatly enhanced by the use of vegetation cultures. With this idea in mind, the referee started twenty-four vegetation experi-

ments with oats and fifteen with sweet potatoes. The oat crops were harvested on August 17, but the pressure of work has been so great that it has been impossible to make the analyses of the different crops. The sweet potato crops are still unharvested. In these vegetation tests portions of the samples of organic nitrogen sent out for analysis were used. It is unfortunate that this work could not be completed prior to this convention, and the referee is forced to defer any further report on it until our next meeting.

The ten samples selected were thought to fairly represent the most common sources of organic nitrogen. The blood, cotton-seed meal, fish, tankage, and hoof represented materials known to possess a high degree of availability, while the wool waste and raw leather were considered to have their nitrogen in a comparatively insoluble form. The three samples of bone were selected to furnish a comparison as to the availability of the nitrogen in the three main classes of ground bone. The first was a button bone which had been thoroughly dried and which contained little fatty matter. The second was a steamed bone of very fine mechanical condition, from which nearly all of the fatty matter had been extracted by the process of steaming, which also destroyed the cellular structure of the bone, and is supposed to render its ingredients more available to the plant. The sample of raw bone consisted of untreated bone; quite a large amount of fatty matter was present.

In sending out the samples the referee requested that four series of determinations should be made:

(1) Total nitrogen by the Kjeldahl method.
(2) The digestibility of the organic nitrogen in pepsin solution.
(3) The digestibility of the organic nitrogen in acid permanganate solution.
(4) The digestibility of the organic nitrogen in alkaline permanganate solution.

(1) Total Nitrogen by the Kjeldahl Method.

Determination of organic nitrogen by Kjeldahl method.

Analysts.	Dried blood.	Cotton-seed meal.	Dried fish.	Tankage.	Hoof meal.	Raw leather.	Wool waste.	Bone sawings.	Steamed bone.	Raw bone.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
R. J. Davidson, Va. Station	13.98	7.23	8.80	6.32	14.11	[1]7.32	2.53	3.91	2.72	4.16
J. P. Street, N. J. Station	14.07	7.39	8.82	6.33	14.12	7.58	2.43	3.92	2.80	4.16
T. C. Trescot, U. S. Dept. of Agr	13.86	7.39	.....	[1]6.09	[1]13.61	7.59	.....	.....	.....	4.18
H. B. Slade, Me. Station	14.19	[1]7.60	.....	6.42	13.95	7.65	.....	.....	.....	4.08
O. W. Knight, Me. Station	14.08	7.46	.....	6.33	13.97	7.40	.....	.....	.....	4.08
L. M. Bloomfield, Ohio Station	.....	7.28	[1]8.57	.....	.....	7.57	2.42	.....	2.80	4.17
W. H. Heileman, Washington Station	.....	.....	8.73	.....	[1]13.58	[1]7.21	2.66	3.95	2.80	.....
Average	14.04	7.33	8.78	6.35	14.04	7.56	2.51	3.93	2.78	4.14

[1] Not included in averages.

COMMENTS OF ANALYSTS.

T. C. Trescot.—The nitrogen was determined in samples 4 and 8 by the regular methods and also by the absolute method of combustion in copper oxid. This method was also used with all the samples. The nitrogen as determined by the moist combustion process agreed very closely with that obtained by the combustion in copper oxid.

L. M. Bloomfield.—I made a number of redeterminations, using 0.5 gram of material

and boiling about six hours. Though the duplicates do not agree as closely as I usually am able to get them, they are much better than the other results sent you. The boiling was evidently the fault. Not having had experience with materials of that kind, I had overlooked the fact that seems to be well established that they need prolonged treatment with the acid in order to get all of the nitrogen. The following are my new determinations:

Sample.	Per cent.	Per cent.
No. 5	7.42	7.70
No. 6	9.10	9.24
No. 9	7.28	7.56

The results of these analyses show quite clearly the necessity of careful manipulation in even such a reliable and well-established method as that of Kjeldahl. As a rule the chief difficulty seemed to be the analyst's inability to secure all the nitrogen in the sample. In every case but one, where there was a wide discrepancy, the results were too low. The low-grade samples, the three bones and the wool waste, occasioned little trouble, but the high-grade materials show variations not to be expected from the hands of experienced analysts. In blood the variation was 0.33 per cent; in cotton-seed meal, 0.37 per cent; in fish, 0.23 per cent; in tankage, 0.33 per cent; in hoof meal, 0.54 per cent, and in leather, 0.41 per cent. It is the belief of the referee that as a rule the low results were caused by insufficient digestion. It is well known that in analyzing such refractory materials as cotton-seed meal, hoof meal, and leather, it is necessary to digest for some time after the liquid becomes colorless. Failure to take this precaution is sure to cause low results.

(2) The Digestion of Organic Nitrogen in Pepsin-hydrochloric Acid Solution.

The method used was substantially the same as that given in the Report of the Connecticut Experiment Station for 1893, page 219.

Table I.—*Organic nitrogen indigestible in pepsin solution.*

Analysts.	Dried blood.	Cotton-seed meal.	Dried fish.	Tankage.	Hoof meal.	Raw leather.	Wool waste.	Bone sawings.	Steamed bone.	Raw bone.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
R. J. Davidson, Va. Station	0.42	0.84	1.96	1.60	6.63	4.51	1.73	0.10	0.45	0.36
	0.52	0.84	1.96	1.68	6.78	4.55	1.79	0.17	0.53	0.45
J. P. Street, N. J. Station	0.49	0.71	1.69	1.69	7.15	5.19	1.61	0.14	0.49	0.36
	0.54	0.88	1.76	[1]1.86	7.22	5.21	1.71	0.20	0.56	0.42
T. C. Trescot, U. S. Dept. of Agr	0.44	0.66	.....	1.49	6.67	[1]3.75	.....	.....	.....	0.39
H. B. Slade, Me. Station	[1]0.14	0.91	.....	[1]2.48	[1]8.85	[1]5.65	.....	.....	.....	[1]1.11
O. W. Knight, Me. Station	0.48	0.66	.....	1.64	6.90	.....	.....	.....	.....	0.29
L. M. Bloomfield, Ohio Station	.....	0.98	2.10	.....	.....	[1]3.85	1.82	.....	[1]0.70	0.49
	.....	[1]1.82	2.17	.....	.....	4.69	1.89	.....	[1]1.05	[1]1.82
W. H. Heileman, Washington Station	.....	.....	1.64	.....	[1]5.90	4.62	1.55	0.03	0.43	.....
	.....	.....	1.66	.....	[1]6.27	4.82	1.64	0.08	0.46	.....
Average	0.48	0.81	1.87	1.62	6.89	4.80	1.72	0.12	0.49	0.39

[1] Not included in averages.

Five of the seven analysts obtained concordant results by this method. Two chemists report results widely divergent from the others. In one case the analyst seemed to have difficulty in securing closely agreeing duplicates, one set of results agreeing closely with those of other analysts, and the other being much too high. The other chemist failed to secure agreement with a chemist working in the same laboratory. It would appear, therefore, that these variations can not be charged to lack of uniformity in the samples or reagents, as the same were doubtless used in both cases, but to that old friend of the referees of this association, the personal factor.

To show the full significance of the results more clearly, the referee has recalculated them on the basis of the per cent of availability.

TABLE II.—*Availability of organic nitrogen in pepsin solution.*

Analysts.	Dried blood.	Cotton-seed meal.	Dried fish.	Tankage.	Hoof meal.	Raw leather.	Wool waste.	Bone sawings.	Steamed bone.	Raw bone.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
R. J. Davidson, Va. Station	96.6	88.5	77.7	74.2	52.3	40.1	29.9	96.7	82.4	90.3
J. P. Street, N. J. Station	96.4	89.2	80.4	73.4	48.9	31.2	33.9	95.7	81.3	90.6
T. C. Trescot, U. S. Dept. of Agr	96.8	91.0	.....	76.5	52.5	[1]50.4	.....	.....	.....	90.6
H. B. Slade, Me. Station	[1]99.1	87.6	.....	[1]61.0	[1]37.0	[1]25.3	.....	.....	.....	[1]73.2
O. W. Knight, Me. Station	96.5	91.0	.....	74.2	50.9	.....	.....	.....	.....	93.0
L. M. Bloomfield, Ohio Station	.....	86.6	75.8	.....	.....	38.0	26.3	.....	[1]68.7	88.2
W. H. Heileman, Washington Station	.....	.....	81.2	.....	[1]56.7	37.6	36.7	98.7	84.2	.....
Average	96.6	89.0	78.8	74.6	52.3	36.7	31.7	97	82.6	90.5
On basis of blood = 68	68	63	55	53	36	26	22	68	58	64
Vegetation cultures	68	74	69	61	67	2	31	.....	60	.....

[1] Not included in averages.

As was pointed out last year, a difference of a few hundredths makes quite a large difference when the results are calculated on this basis. For instance, the extreme variation in the cotton-seed meal from 86.6 per cent to 91 per cent is caused by a difference of but 0.32 per cent, while the variation in the leather of 8.9 per cent is due to a difference of but 0.57 per cent. When considered from this standpoint the results are remarkably uniform and show that this method will give concordant results in the hands of different analysts in different laboratories. The value of the method depends, however, on how far it is an interpreter of the actual results secured by the growing plant. As pointed out above, the referee was unable to finish his vegetation tests in time to secure data on this point. However, as the materials used were of average grade, except possibly the wool waste, which was an unusually good sample of that product, a rough comparison may be made with vegetation tests on the same class of materials by other workers. Using the results obtained by Dr. Jenkins, we find a fair agreement in all classes of goods except in the case of the hoof meal, whose vegetation test availability is 67 but whose pepsin availability is but 36. This fact merely emphasizes what has been noted by other workers, that the pepsin digestibility of hoof meal is no measure of its availability to the plant.

(3) THE DIGESTION OF ORGANIC NITROGEN IN PERMANGANATE OF POTASH SOLUTION.

Method in acid solution.—Weigh 1 gram of material into a 500 cc flask, add a little paraffin and 100 cc of permanganate solution (consisting of 16 grams of potassium

permanganate and 100 cc of concentrated c. p. sulphuric acid to the liter). Connect with distilling apparatus and heat at a low temperature just below boiling for one hour. Then add 50 cc of saturated sodium hydrate solution, distill for thirty minutes, and titrate as usual.

Method in alkaline solution.—Weigh 1 gram of material into a 500 cc flask, add a little paraffin and 100 cc of permanganate solution (consisting of 16 grams of potassium permanganate and 300 cc of saturated sodium hydrate solution to the liter). Connect as before and heat below boiling for one hour. Then increase the temperature and distill for thirty minutes as above.

TABLE III.—*Availability of nitrogen in alkali permanganate.*

Analysts.	Dried blood.	Cotton-seed meal.	Dried fish.	Tankage.	Hoof meal.	Raw leather.	Wool waste.	Bone sawings.	Steamed bone.	Raw bone.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
R. J. Davidson, Va. Station	42.2	38.1	49.8	55.3	46.6	24.6	69.3	7[illegible].9	78.8	76.1
J. P. Street, N. J. Station	26.6	23.9	23.9	29.4	25.2	9.3	33.9	54.4	49.3	44.0
T. C. Trescot, U. S. Dept. of Agr.	60.9	64.3	.....	76.1	67.9	43.3	.....	.....	.....	82.6
H. B. Slade, Me. Station	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
O. W. Knight, Me. Station	31.8	31.8	.....	48.5	39.6	17.2	.....	.....	.....	78.0
L. M. Bloomfield, Ohio Station	.....	30.6	32.8	.....	.....	19.4	58.6	.....	68.0	62.6
W. H. Heileman, Washington Station	.....	.....	29.8	.....	28.7	12.6	50.2	68.2	63.7	.....

TABLE IV.—*Availability of nitrogen in acid permanganate.*

Analysts.	Dried blood.	Cottonseed meal.	Dried fish.	Tankage.	Hoof meal.	Raw leather.	Wool waste.	Bone sawings.	Steamed bone.	Raw bone.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
R. J. Davidson, Va. Station	42.9	42.3	53.5	64.4	46.4	29.1	62.9	79.9	77.7	77.7
J. P. Street, N. J. Station	38.7	36.6	48.9	62.4	42.3	21.2	61.8	74.3	73.4	71.3
T. C. Trescot, U. S. Dept. of Agr.	53.1	65.8	.....	66.1	51.7	55.3	.....	.....	.....	74.4
H. B. Slade, Me. Station	35.2	32.7	.....	58.0	49.0	22.5	.....	.....	.....	69.3
O. W. Knight, Me. Station	34.7	31.1	.....	56.4	33.5	17.6	.....	.....	.....	70.0
L. M. Bloomfield, Ohio Station	.....	30.6	31.9	.....	.....	18.5	61.4	.....	70.5	54.1
W. H. Heileman, Washington Station	.....	.....	40.2	.....	35.7	16.1	38.6	63.1	61.9	.....

There is an utter lack of agreement in the results by these two methods, especially where the alkaline permanganate is used. The referee was unable to test this method thoroughly before sending out the instructions, and in this way certain possible sources of error escaped his attention which would have been otherwise noticed. In the work with this method last year, Mr. R. J. Davidson and the referee were

the only chemists who reported results, and their work was upon a limited number of materials. Their results agreed quite closely and compared well with the vegetation tests in the same samples. The acid solution appeared to be particularly promising.

After making a number of tests this year, it became evident to the referee that the method as suggested was a difficult one to control. The directions provided that during the digestion the solutions should be heated just below boiling. This was found to be almost an impossibility, and it was very difficult to maintain uniform conditions in two different sets of tests. It is more than likely, therefore, that this inability to control the temperature and keep it uniform is in a large measure responsible for the poor agreement of the results. A study of the table shows, however, that notwithstanding this failure to agree with each other, the results of the different analyses, if taken separately, show a very decided uniformity in the relative availability of the different organic forms. The three forms of bone were found to be the most available, while the others followed in this order: tankage, wool waste, fish, hoof meal, blood, cotton-seed meal, and leather. These results were far from satisfactory, as it was known that with the exception of the leather the order of their availability should be almost reversed.

AVAILABILITY OF NITROGEN TAKEN IN EQUAL AMOUNTS.

It then occurred to the referee that the difficulty might be obviated were the same amount of organic nitrogen used in each determination instead of the same amount of material. Accordingly a new series of determinations was made, where an amount of organic nitrogen equivalent to that contained in one gram of cotton-seed meal was taken in each case. The results of these analyses follow:

TABLE V.—*Availability of nitrogen in equal amounts.*

Substances.	Amount taken.	Availability.	Amount taken.	Availability.
	Grams.	*Per cent.*	*Grams.*	*Per cent.*
Dried blood	1	26.6	0.5	26.9
Cotton-seed meal	1	23.9	1.0	21.0
Dried fish	1	23.9	.8	23.7
Tankage	1	29.4	1.2	26.7
Hoof meal	1	25.2	.5	32.9
Raw leather	1	9.3	1.0	9.2
Wool waste	1	33.9	3.0	25.1
Bone sawings	1	54.4	1.9	31.1
Steamed bone	1	49.3	2.6	26.8
Raw bone	1	44.9	1.8	30.5

The relation shown by these results was much more in accord with the vegetation tests, and the results appeared to determine quite closely the relative availability of the different forms.

The failure of the method in the hands of different analysts led the referee to attempt to devise some method whereby the conditions could be controlled more accurately. It was suggested by the Maine Station that a 3 per cent neutral solution of permanganate be used and that the digestion take place on a steam bath. Acting on this suggestion, the referee adopted the following line of treatment, and secured very promising results.

Method.—Weigh 1 gram of material into a 500 cc Erlenmeyer flask; add 100 cc of neutral 3 per cent permanganate of potash solution, and digest on a steam bath for thirty minutes, shaking occasionally to moisten any particles of the material adhering to the sides of the flask. Add 200 cc of cold water, and filter and wash three or four

times, using from 125 to 150 cc of water. Determine the total nitrogen in the undigested residue by the ordinary Kjeldahl method.

In the first tests by this method an attempt was made to filter the solutions while still hot, but it was found that aside from the difficulty of handling a hot solution the great concentration of the liquid made the filtration very tedious. It was demonstrated by comparative results, as will be noted later, that dilution of the solution gave even better results, and this practice was employed in all subsequent tests by this method.

In the following table are also incorporated some tests made by Mr. H. B. Slade, of the Maine Station. In his report Mr. Slade did not specify the method of filtration used, and it is assumed that it was carried on while the solutions were still hot. The calculations are made on the basis of per cent of availability.

TABLE VI.—*Availability of nitrogen in equal amounts.*

Substances.	Street.	Slade.	Average.
	Per cent.	*Per cent.*	*Per cent.*
Dried blood	95.3	94.3	94.8
Cotton-seed meal	92.5	89.4	91.0
Dried fish	92.8	.....	92.8
Tankage	94.5	95.0	94.8
Hoof meal	93.9	91.3	92.6
Raw leather	64.2	26.5	45.3
Wool waste	90.8	.....	90.8
Bone sawings	91.6	.....	91.6
Steamed bone	92.1	.....	92.1
Raw bone	86.2	94.1	90.2

A study of the table shows a very marked difference between the availability of the leather and the other materials, the availability ranging from 94.8 per cent in the case of the blood to 45.3 per cent in the case of the leather. The wool waste shows an availability of 90 8 per cent, higher than one would expect, but, as was pointed out earlier in the report, this sample was an unusually good product and probably does not fairly represent the average wool waste on the market. It is impossible to draw any positive conclusions from these results without vegetation tests as a basis of comparison, but it is believed that further study of this method, possibly with variations in the strength of the permanganate solution, may point out a chemical method which will secure results in close accord with vegetation cultures.

RECOMMENDATIONS.

(1) That the Ulsch-Street method for nitric nitrogen be made an official method.

(2) That the study of the Ulsch-Kjeldahl and Ulsch-Gunning methods for total nitrogen in the presence of nitrates be continued next year.

(3) That the permanganate of potash method for determining the availability of organic nitrogen be further studied next year, with especial reference to materials of known inferiority.

COMMITTEE ON RECOMMENDATIONS—DISCUSSION ON NITROGEN.

Upon the conclusion of Mr. Street's paper the chair appointed the following committee upon recommendations: Messrs. Bartlett, Wheeler, Scovell, Van Slyke, and Hite.

Mr. WINTON. I would like to say a word with regard to this Ulsch-

Gunning or Fassbänder-Gunning method, as we have been accustomed to call it. This method has given very good results indeed at our station. We have tried it during the past year on every nitrate-containing fertilizer that has come into our laboratory, and it has given very good satisfaction on all samples, with a few exceptions. You will notice that the samples the referee sent out contained in no case more than $1\frac{3}{4}$ per cent of nitric nitrogen. On such samples of fertilizers the method is perfectly satisfactory. The time of digestion, in our experience, has been a little more than half that required in the modified Kjeldahl method. It seems to meet every requirement in the case of such samples, but when the percentage gets up to 3, 4, or 5, or higher, it is not at all accurate. We lose several tenths of a per cent of nitrogen. This point was brought out by Mr. Ogden, of our station, some years ago in connection with the Ulsch method. He found that 2 grams of iron would not give him theoretical results on pure salts where the percentage was 15 or 16; that is, when the nitric nitrogen present was as much as .07 gram. In such cases it required more iron. Of course, it naturally followed in this Ulsch-Gunning method, which is merely the Ulsch method carried a little further, that a larger quantity of iron was necessary, so that in these exceptional samples, with more than 3 per cent of nitric nitrogen, we were obliged to use more iron. We did it reluctantly, because the extra amount very materially interferes with the distillation and causes bumping too early in the process. We made numerous experiments with various quantities of acid to see if it were not possible to get along with a smaller amount, but we were not successful. Working on pure nitrate of potash in half-gram portions, in no case was I able to get over 13.30 per cent, whereas the theoretical per cent was 13.80, or a loss of one-half of 1 (0.5) per cent. So it seems necessary in such cases to use a larger quantity of iron. Now, I think 3 grams of iron will meet every possible case likely to be encountered in the analysis of mixed fertilizers, but our experiments show that when the percentage of nitrogen is as high as 7, even that quantity is insufficient. But such fertilizers are not likely to be encountered. I think, however, it will be well if in the ensuing year the point can be taken into consideration whether or not it is desirable to modify the method in such a way as to give results for fertilizers containing high percentages of nitrogen.

I want to say a word as to the availability of nitrogen in different solutions. It seems to me that the pepsin solution tells a better story than any of these other solutions. For example, you are probably familiar with the work that has been done at the Connecticut Station on a variety of substances, and if you remember those results, they showed that while dried blood and cotton-seed meal did not give as high as 96.89 per cent available nitrogen, nevertheless, with the pepsin solution, a better clew to the available organic nitrogen in these substances was obtained than by any other reagent that had been used.

The point I wish to make is, that although this pepsin solution will not coincide, percentage for percentage of available nitrogen, at all closely with actual results obtained from pot experiments, yet it gives much better results than other solutions.

With anything that has somewhere about 80 or 90 per cent of available nitrogen the pepsin solution will show a very high percentage when tested with pot experiments. When we came to wool waste and raw leather we found the pepsin solution in a pot experiment gave much lower results than that. Now, Dr. Jenkins has proved that raw leather contains not over 6 to 7 per cent available nitrogen. In experiments conducted at our station for two years on raw leather, we found just the same thing, whereas the availability is as high as 36 per cent. So we established the rule that anything under 50 per cent, using the pepsin solution, would be considered nonavailable, but anything running above 50 per cent, with the pepsin solution, would be available. The pepsin solution, considered in that way, gave us a very good idea of the relative availability of different organic matters.

The PRESIDENT. I did not notice that you said anything with reference to hoof meal.

Mr. WINTON. There was one case where it did not work. I think hoof meal was proved by pot experiments to be quite valuable. Taken in a general way, the pepsin solution seemed to be the best thing we could employ for the purpose. I think it does give us a very good clew to the available organic nitrogen.

Mr. STREET. The chief reasons for seeking another method for determining the availability of nitrogen were, first, that in the case of this hoof meal we got entirely too low availability by the pepsin, and the leather was entirely too high, and, secondly and chiefly, that it takes a great amount of time. It requires twenty-four hours digestion.

Mr. LINDSEY. It seems to me that the acid permanganate and alkali permanganate did not tell a very straight story.

Mr. STREET. They are no good at all.

Mr. Ewell offered the following resolution:

Resolved, That henceforth in reporting results of trials of methods the following data are to be included:

Number of determinations by each analyst.
Maximum of determinations by each analyst.
Minimum of determinations by each analyst.
Average of determinations by each analyst.

The attention of the referee to be called to the fact that the general averages should be calculated from the sum of all the determinations and not from the sums of averages for individual analysts.

Referred to the committee on recommendations.

REPORT OF COMMITTEE ON RECOMMENDATIONS.

The report of the committee on recommendations was then called for and read by Mr. Bartlett.

The committee recommends that—

1. The Ulsch-Street method for determination of nitric nitrogen, as recommended by referee, be made an official method.
2. That the Ulsch-Kjeldahl or Ulsch-Gunning method be further investigated.
3. That the referee and members of the association devote what time there may be available to devising and perfecting a rapid method of determining available nitrogen in mixed fertilizers, and that the referee call the attention of members to the subject in sending out his directions for work.

Mr. Van Slyke moved that the first recommendation be adopted; viz, that the Ulsch-Street method for nitrogen be made an official method. Adopted.

Mr. Huston moved that the second recommendation be adopted; viz, that the Ulsch-Kjeldahl or Ulsch-Gunning method be made the subject of further study. Adopted.

Mr. Van Slyke moved the adoption of the third recommendation; viz, that the study of a rapid method for determining available nitrogen be earnestly commended to both referees and members of the association.

Mr. WILEY. I have no vote on this question, but perhaps I may say that rapidity is not the only thing to be considered. Would it not be well to say "a rapid and reasonably reliable method?"

The Chair reminded the association that only those members actually concerned with fertilizer control work were entitled to vote upon these matters of fertilizer analysis.

Mr. Persons asked if the association could not allow those chemists who were in charge of fertilizer work at stations, though not actually official State chemists, to vote upon these questions. The Chair referred Mr. Persons to the history of this restriction on voting, stating that it had been made upon good grounds, and that if he would look up the records he would probably not be disposed to criticise this clause.

The recommendation was thereupon adopted.

Mr. BARTLETT. We have no further recommendation with regard to this matter of available nitrogen.

After some discussion, it was agreed that the hours for meeting should be from 9.30 a. m. to 12.30 p. m., and from 2 to 4.30 p. m.

The report on potash was read by Mr. Winton, as follows:

REPORT ON POTASH.

By A. L. WINTON, *Referee*, and C. H. JONES, *Associate Referee*.

The work of the present year has been carried out with the cooperation of the following chemists: Mr. G. E. Patrick, United States Department of Agriculture, Division of Chemistry, Washington, D. C.; Mr. A. M. Peter, Kentucky Experiment Station, Lexington, Ky.; Mr. F. B. Bomberger, Maryland Agricultural College, College Park, Md.; Mr. A. W. Blair, North Carolina Experiment Station, West Raleigh, N. C.; Mr. B. L. Hartwell, Rhode Island Experiment Station, Kingston, R. I.; Mr. F. S. Shiver, South Carolina Experiment Station, Clemson, S. C.

The subjects which have been investigated are:

(1) The Stassfurt methods for determination of potash.

(2) The "optional" method, acidulating with hydrochloric acid before precipitating the sulphuric acid.

(3) The determination of potash by the Lindo-Gladding method, evaporating directly with platinum solution, without previous removal of lime.

(4) Quantities of platinum solution necessary for determinations in German potash salts.

Preparation of Materials.

A sufficient quantity of the materials used in the study of potash methods in 1896 remained for the determinations of the present year. These materials, as was fully described in the report on potash last year, consisted of pure potassium chlorid, pure potassium sulphate, and mixtures representing the impurities in each of the four manure salts commonly sold in the United States. The "impurities" were distributed in vials in quantities corresponding to 10 grams of the commercial salts of average composition. The total contents of each vial were boiled by each analyst with water, made up to a definite volume and filtered. By mixing, in the proper proportions, aliquots of this solution with weighed portions of the pure potash salts, solutions for analysis were obtained which represented definite quantities of the commercial salts.

Details as to quantities are given in the following table:

Quantities of samples.

Impurities.	Solution of "impurities" made up to—	Equivalent to 0.5 gram of the Stassfurt salt.	
		Solution of "impurities."	Pure potash salt.
	cc.	*cc.*	*Gram.*
Muriate of potash	500	25	0.42 KCl
Sulphate of potash 90 per cent	400	20	.46 K_2SO_4
Sulphate of potash and magnesia	400	20	.26 K_2SO_4
Kainit	125	6¼	.14 K_2SO_4

In making comparative determinations by the Lindo-Gladding method, as applied to kainit, samples of the commercial salt, as well as artificial mixtures, have been used.

(1) The Stassfurt Methods.

Through the courtesy of Dr. B. von Herff, of the New York office of the potash syndicate, copies of the description of the Stassfurt methods, printed in the German language, were secured for distribution among the chemists participating in the investigation of the methods.

The following translation was specially prepared by Dr. von Herff for publication in this report. The footnotes were communicated by chemists of the syndicate:

Description of methods of analysis agreed upon and adopted by the Stassfurt potash industry.

A.—*Method for the Determination of Potash in the Concentrated Salts.*

(1) *Muriate of potash.*

(*a*) *Determination of potassium chlorid.*

Of the finely ground substance, 7.6405 grams are dissolved and the solution made up to 500 cc. If the sample contain more than 5 per cent of sulphuric acid (SO_3) it

will be necessary to transform the sulphates to the corresponding chlorin compounds by precipitating with acidulated solution of barium chlorid. Twenty cubic centimeters of the filtrate (= 0.3056 gram substance) are evaporated with a 5 cc platinum chlorid solution upon the water bath in a shallow porcelain dish of about 10 cm diameter. The evaporation is facilitated by stirring, and is carried to the point where the thick liquid rapidly crystallizes upon cooling.[1] The dry residue is finely ground with a glass pestle, then stirred up with 20 cc of alcohol and poured upon the filter, which has previously been weighed (while warm) after drying at 120° to 130° C. One hour's drying will usually be found necessary. The filter should first be moistened with alcohol, and care should be taken not to pour any of the liquid upon the rim of the filter. The filtration may be accelerated by the weak action of a suction pump.[2] The potassium platinum chlorid can be readily and completely washed upon the filter. The precipitate is freed from the alcohol as much as possible by suction and by pressing between filter papers; it is then dried at 120° to 130° until its weight is constant. This will require about twenty minutes. One milligram potassium platinum chlorid corresponds to 0.1 per cent potassium chlorid.

(b) Determination of chlorid of sodium.

Twelve and a half grams chlorid of potassium are dissolved in a 250 cc flask by boiling with 25 cc water, to which a little potassium carbonate has been added in order to transform the compounds of magnesium and calcium into carbonates. The flask is filled up to the mark with absolute alcohol. One hundred cubic centimeters of the filtrate (= 5 grams of the substance) are evaporated to dryness in a porcelain or platinum dish, after a few drops of the concentrated hydrochloric acid have been added to transform the potassium carbonate, which might be present, into potassium chlorid; the residue is heated to a low redness and weighed. In this mixture of the chlorids of potassium and sodium, either chlorid of potassium is determined after the common method with platinum chlorid and sodium chlorid, calculated from the difference, or it is determined by titrating the mixed alkalies with one-tenth normal silver solution.

(c) Determination of chlorid of magnesium.

Twenty-five grams of chlorid of potassium are dissolved in a 500 cc flask, and 10 cc of normal potash solution are added. The contents of the flask are made up to the mark, and 50 cc of the filtrate are titrated with one-tenth normal sulphuric acid. The soluble compounds of calcium present do not influence the result.

(2) Determination of sulphate of potassium and sulphate of potassium magnesium.

(a) Determination of potassium sulphate.

Dissolve 8.9235 grams of the finely ground substance in a 500 cc flask with about 350 cc boiling water, to which 20 cc of concentrated hydrochloric acid have been added. The sulphuric acid is precipitated by slowly adding barium chlorid solution (from a burette with glass cock) to the boiling liquid. The precipitation of the sulphuric acid is completed if the clear solution do not become turbid by the addition of a small fragment of barium chlorid. This is a very accurate test. If an excess of barium chlorid have been added, it must be precipitated with sulphuric acid. After completing the precipitation and cooling, the flask is filled up to the mark and 20 cc of the filtrate (= 0.357 gram of the substance) are treated as usual with 5 cc of platinum chlorid solution. One milligram K_2PtCl_6 corresponds to 0.1 per cent K_2SO_4. Three-tenths of 1 per cent are to be added to the result obtained in the analyses of sulphate of potash, while in the analyses of sulphate of potash-magnesia no correction is necessary.[3]

(3) Determination of potassium chlorid and potassium sulphate in calcined manure salts.

(a) Determination of potassium chlorid and potassium sulphate.

Dissolve 15.281 grams of the sample (for the determination of muriate of potassium) or 17.847 grams (for the determination of sulphate of potassium) in a 500 cc

[1] It is advantageous to evaporate the solution to complete dryness in case it contains potassium platinum chlorid and sodium platinum chlorid only, because the sodium platinum chlorid is thereby rendered free of all water, and in this form it is more soluble in absolute alcohol, or in alcohol of 96 per cent.

[2] For the chemist with little experience, it is advisable to grind the precipitate once or twice with fresh quantities of alcohol after the first washing liquid has been poured off.

[3] In order to obtain accurate results it is absolutely necessary to follow the described method in all details. Results will be unreliable and more troublesome corrections will be needed if the liquid be either more diluted or more concentrated, or if the sulphuric acid be precipitated from a solution which is either very slightly acid or even neutral, or if the boiling during precipitation be interrupted.

flask with water, to which 10 cc of concentrated hydrochloric acid have been added. The flask is filled up to the mark, the contents filtered, and 250 cc (=7.6405 and 8.9235 grams, respectively) of the filtrate are treated in a 500 cc flask with barium chlorid solution in the same manner as in the determination of potassium sulphate.

B.—*Method for Determination of Magnesium Sulphate in Kieserit.*

Ten grams of the finely ground substance are boiled for at least an hour in a 500 cc flask about two-thirds full of water. After cooling, from 50 to 60 cc of double normal potash solution and 20 cc (1:10) neutral potassium oxylate solution are added. The flask is filled up to the mark, and the liquid filtered, after standing for a quarter of an hour. The excess of alkali in the filtrate is titrated with one-tenth normal sulphuric acid, and 0.2 per cent are to be added to the sulphate of magnesia obtained.

C.—*Methods of Analysis of Crude Potash Salts (Carnallit, Kainit, Sylvinit, and Bergkieserit).*

(*1*) *Preparation of sample.*

It is recommended to grind a large sample of at least 0.5 kg in weight in a mill or mortar, in order to avoid differences resulting from coarse grinding.

(*2*) *Determination of potash by the precipitation method.*

Dissolve 35.70 grams of kainit or sylvinit, or 30.56 carnallit or bergkieserit, in a 500 cc flask, with about 350 cc boiling water, to which 10 cc hydrochloric acid have been added. After cooling, the flask is filled up to the 500 cc mark, 50 cc of the filtrate are precipitated in a 200 cc flask with barium chlorid in the usual manner, and 20 cc (=0.3570 gram, 0.3056 gram substance, respectively) are evaporated with 5 cc platinum chlorid solution, and further treated according to the common method.[1]

(*3*) *Complete analysis of the crude potash salts.*

For this purpose, 100 grams of the substance are dissolved by boiling with about 500 cc water. The solution is filtered, the residue and filtrate washed, and the filtrate and wash waters made up to 1 liter. A portion of the liquid serves for the determination of sulphuric acid, according to the common method, by precipitating with barium chlorid; another portion serves for the determination of lime and magnesia. For the determination of the alkaline chlorids 100 cc of the solution (=10 grams of the substance) are acidulated with hydrochloric acid, heated to boiling, and the sulphuric acid completely precipitated with barium chlorid in a 500 cc flask, avoiding an excess of the barium solution. Fifty cubic centimeters of this liquid (=1 gram substance) are evaporated to dryness in order to drive off the hydrochloric acid, and thereafter magnesium chlorid is decomposed by igniting with oxalic acid. After the ignition, the residue is moistened with a little ammonium carbonate in order to transform the calcium oxid into calcium carbonate. In the mixture of the chlorids of the alkalies, which are now entirely free from lime and magnesia, potassium chlorid is determined by evaporating with 10 cc platinum chlorid solution. The weight of the sodium chlorid is obtained by subtracting potassium chlorid from the mixed alkalies.

In calculating the results obtained from kainit and sylvinit, the sulphuric acid combined with calcium is first subtracted from the total amount of soluble sulphuric acid. The remainder of the sulphuric acid is calculated to potassium sulphate and magnesium sulphate, according to the molecular proportion in which these salts are present in kainit and schoenit. If, after this calculation, some potassium remain uncombined with sulphuric acid, it is to be reckoned as potassium chlorid. Likewise the remainder of the magnesia, uncombined with sulphuric acid, is to be reckoned as magnesium chlorid. The result of the calculation will show how much potassium is present on one hand in the form of kainit, K_2SO_4, $MgSO_4$, $MgCL_2 + 6H_2O$, and schoenit, K_2SO_4, $MgSO_4 + 6H_2O$, and, on the other hand, in the form of potassium chlorid. Sodium is reckoned as chlorid of sodium.

In carnallit and bergkieserit calcium oxid is calculated to sulphate of calcium, and the remainder of the sulphuric acid, not combined with calcium, is to be reckoned as sulphate of magnesium. Magnesia uncombined with sulphuric acid is to be reckoned as magnesium chlorid.

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[1] To be more particular, it is recommended here to take a rather larger quantity of platinum chlorid, say 6 to 7 cc, and also to pay attention to the footnote No. 2, page 31.

D.—*Determination of Water.*

The content of water in chlorid of potassium and in the other concentrated salts is determined by heating 10 grams of the sample in a covered platinum crucible for about ten minutes, with a small flame to dark-red heat. In the analysis of salts which are rich in magnesium chlorid care should be taken to avoid the decomposition of that constituent. This can be effected by covering the substance with freshly ignited lime,[1] or else the loss of chlorin should be determined by double titration, paying attention at the same time to the absorption of oxygen. The loss in weight is to be subtracted from the result obtained in the determination of water.

APPENDIX.

(1) *Preparation of the solution of platinum chlorid.*

The recovery of the platinum from the alcoholic wash liquids containing potash salts, and also from the precipitates of potassium platinum chlorid, is carried out in a large porcelain dish upon the water bath. The alcoholic liquid, after being diluted with about one-third of water, is heated to boiling, sodium carbonate is added, and at the same time the precipitates of potassium platinum chlorid are put in the reducing liquid in small portions at a time. The heating upon the water bath should be continued until the alkaline liquid, covering the precipitated platinum, is perfectly clear. Its color should be only of the faintest yellow. After pouring off the liquid, the reduced platinum is cleansed by boiling with hydrochloric acid and water. It is then dried upon the water bath and ignited for the purpose of destroying organic compounds. It is a good plan to heat the finely ground platinum once with pure concentrated nitric acid, which is to be poured off. The platinum is dissolved in a large porcelain dish upon the water bath by first heating with about four times its weight of pure concentrated hydrochloric acid, to which nitric acid (in proportion of four parts of hydrochloric acid to one of nitric acid) is gradually added. After the solution is completed, it is to be evaporated until a drop taken out with a glass rod solidifies on cooling. The solution of platinum chlorid is allowed to cool, and will then form a crystalline cake, which is dissolved in water, filtered, and diluted, so that 1 gram of platinum is contained in 10 cc of solution (sp. gr. 1.18). Special care must be taken that the solution is free from subchlorid of platinum as well as from nitrogen compounds. Platinum subchlorid may be converted into platinum chlorid by means of fuming hydrochloric acid and a little nitric acid. The nitrogen compounds are destroyed by repeated additions of hydrochloric acid and water during evaporation. If platinum scraps have been used for making platinum chlorid, it should be freed from iridium by precipitating the platinum chlorid solution with ammonium chlorid and again reducing the precipitate. The testing of the purity of the platinum chlorid solution is most conveniently performed by using potassium chlorid prepared from chemically pure materials.

(2) *Preparation of barium chlorid solution.*

One hundred and twenty-two grams of crystallized barium chlorid are dissolved with water and 50 cc concentrated hydrochloric acid and diluted to 1,000 cc.

(3) *Alcohol.*

Alcohol for washing the platinum precipitate should be of at least 96 per cent strength.

(4) *Filter.*

For filtering potassium platinum chlorid, the Swedish filter Munktell 1 F. is best suited.[2]

The following special instructions were sent out by the referees:

INSTRUCTIONS FOR TESTING THE METHODS.

The analyst should first familiarize himself with the method of precipitating $BaSO_4$, working on solutions prepared as directed from any samples of real Stassfurt salts.

[1] Or with lead oxid.

[2] Or else the Gooch crucibles with asbestos may be employed for the purpose.

Muriate of potash.—Weigh out accurately from a weighing tube about 0.25 gram, KCl, c. p., add 15 cc muriate of potash "impurities" and 5 cc of water. Evaporate with 5 cc of platinum solution and proceed otherwise according to A, 1, a. Make blank on reagents and "impurities."

Sulphate of potash.—Weigh out exactly 3.2838 grams K_2SO_4, c. p. Transfer to a 200 cc flask, add 143 cc sulphate of potash "impurities" and 8 cc HCl concentrated. After precipitating $BaSO_4$, as directed under A, 2, make up to 200 cc, filter through dry paper and proceed with 20 cc as further directed.

Sulphate of potash and magnesia.—Weigh out exactly 1.8561 grams K_2SO_4, c. p. Transfer to a 200 cc flask, add 143 cc sulphate of potash and magnesia "impurities" and 8 cc HCl concentrated and proceed as with sulphate of potash.

Kainit.—Weigh out exactly 0.9996 gram K_2SO_4, c. p. Transfer to a 200 cc flask, add 45 cc kainit "impurities," 1 cc HCl concentrated, and 4 cc water. Precipitate SO_3 and proceed as directed under C, 2.

Make blank determinations on reagents (not impurities) used in the analysis of the preceding three salts as follows: Place in a 200 cc flask the quantities of $BaCl_2$ solution and HCl which were used in the actual analysis. Add water so the bulk of liquid will be 150 cc and heat to boiling. Add dropwise slight excess of pure H_2SO_4 (which should leave no residue on evaporation); allow to stand some time, make up to the mark, and filter through dry paper. Evaporate 20 cc, drive off H_2SO_4, add water and 1 cc HCl concentrated. Proceed with evaporation with platinum solution, treatment with alcohol, etc., as in the actual analysis.

For filtering and weighing the K_2PtCl_6 use Gooch crucibles in all cases.

In testing the method for muriate of potash, it was possible to weigh out the KCl needed for each determination and add to it the proper quantity of the solution of "impurities," thus avoiding the errors and waste of materials incident to making up a larger amount to a definite volume and removing aliquot portions.

This could not be done in the other cases, but a saving of materials was effected by using smaller quantities and making up to correspondingly smaller volumes. The aliquot portions, after precipitation of $BaSO_4$, in all cases represented the same quantities of material as they would if the normal quantities had been used.

The determination of the potash in the reagents and "impurities" together represented certain difficulties in the case of the salts containing sulphates, and as the quantities in the "impurities" were too small to be considered, only blanks on the reagents were made.

The analytical data obtained by the several analysts, together with calculated results, are given in Tables I and II.

Before converting to KCl or K_2SO_4, the total K_2PtCl_6 found in each determination was first corrected for the amount found in the average of the blank determinations by the same analyst. As directed in the description of the method, the old conversion factors (0.3056 to KCl and 0.3570 to K_2SO_4) were employed. In the case of sulphate of potash, 90 per cent of the weights of K_2SO_4 found were corrected by adding 0.0011 gram, corresponding to the correction of 0.3 per cent, which, according to the directions, should be made in the analysis of this salt.

TABLE I.—*Determination of potassium chlorid by the Stassfurt method.*

[Old factors.]

Substances and analysts.	KCl (dry) taken.	K_2PtCl_6 total found.	K_2PtCl_6 reagents and "impurities."	KCl found.	KCl error.	K_2O error.
Potassium chlorid, c. p.:	*Gram.*	*Gram.*	*Gram.*	*Gram.*	*Gram.*	*Gram.*
A. W. Blair	0.2489	0.8225	0.0060	0.2495	+0.0006	+0.0004
	.2489	.8236	.....	.2499	+ .0010	+ .0006
	.2489	.8202	.....	.2488	— .0001	— .0001
	.2489	.8176	.....	.2480	— .0009	— .0005
Muriate of potash. (KCl, c. p. + impurities):						
G. E. Patrick	.2400	[1].7843	.....	.3397	— .0003	— .0002
	.2404	[1].7848	.....	.2398	— .0006	— .0004
	.3469	[1]1.1329	.....	.3462	— .0007	— .0004
	.4945	[1]1.6156	.....	.4937	— .0008	— .0005
	.2787	[1].9102	.....	.2782	— .0005	— .0003
	.2832	[1].9215	.....	.2816	— .0016	— .0010
	.2953	[1].9662	.....	.2953	.0000	.0000
	.2815	[1].9202	.....	.2812	— .0003	— .0002
	.2551	[1].8401	.....	.2567	+ .0016	+ .0010
	.2701	[1].8820	.....	.2695	— .0006	— .0004
	.2461	[1].8030	.....	.2454	— .0007	— .0004
	.2836	[1].9267	.....	.2832	— .0004	— .0003
A. M. Peter	.2489	.8157	.0005	.2491	+ .0002	+ .0001
	.2489	.8161	.....	.2492	+ .0003	+ .0002
F. B. Bomberger	.2421	.7953	.0053	.2414	— .0007	— .0004
	.2424	.7976	.....	.2421	— .0003	— .0002
	.2516	.8281	.....	.2514	— .0002	— .0001
	.2573	.8459	.....	.2569	— .0004	— .0003
A. W. Blair	.2489	.8312	.0095	.2511	+ .0022	+ .0014
	.2489	.8320	.....	.2514	+ .0025	+ .0016
F. S. Shiver	.2495	.8195	.0020	.2498	+ .0003	+ .0002
	.2511	.8247	.....	.2514	+ .0003	+ .0002
C. H. Jones	.2556	.8370	.0045	.2544	— .0012	— .0008
	.2556	.8400	.....	.2553	— .0003	— .0002
	.2556	.8374	.....	.2545	— .0011	— .0007
	.2556	.8394	.....	.2551	— .0005	— .0003

[1] Corrected by blanks with impurities and reagents.

TABLE II.—*Determination of potassium sulphate by the Stassfurt method.*

[Old factors.]

Substances and analysts.	K_2SO_4 taken.	K_2PtCl_6 total found.	K_2PtCl_6 from reagents.	K_2SO_4 found.	K_2SO_4 error.	K_2O error.
Potassium sulphate c. p.:	*Gram.*	*Gram.*	*Gram.*	*Gram.*	*Gram.*	*Gram.*
A. W. Blair	0.3284	0.9532	0.0088	0.3383	+0.0099	+0.0054
	.3284	.9529	.0096	.3381	+ .0097	+ .0052
	.3284	.9553	.0081	.3390	+ .0106	+ .0057
	.3284	[1].9081	.....	[1].3222	— .0062	— .0033
	.3284	[2].9083	.....	[2].3222	— .0062	— .0033
	.3284	[3].9110	.....	[3].3232	— .0052	— .0028
	.3284	[4].9025	[4].0076	[4].3206	— .0078	— .0042
	.3284	[4].9023	.....	[4].3205	— .0079	— .0042
C. H. Jones	.3570	.9920	.0030	.3542	— .0028	— .0015
	.3570	.9950	.....	.3552	— .0018	— .0010
Sulphate of potash, 90 per cent (K_2SO_4 c. p. + impurities):						
G. E. Patrick, solution No. 1	.3284	[5].9214	.....	.3300	+ .0016	+ .0009
	.1642	[5].4532	.....	.1629	— .0013	— .0007
G. E. Patrick, solution No. 2 [7]	.3284	[5].9095	.....	.3258	— .0026	— .0014
	.3284	[5].9132	.....	.3271	— .0013	— .0007
	.3284	[5].9145	.....	.3276	— .0008	— .0004
	.3284	[5].9141	.....	.3274	— .0010	— .0005
	.3284	[5].9217	.....	[6].3301	+ .0017	+ .0009
	.3284	[5].9149	.....	.3277	— .0007	— .0004
	.3284	[5].9142	.....	.3274	— .0010	— .0005
A. M. Peter	.3284	.9149	.0017	.3271	— .0013	— .0007
F. B. Bomberger	.3284	.9193	.0063	.3270	— .0014	— .0008
	.3284	.9208	.....	.3266	— .0018	— .0010
	.3284	.9211	.....	.3277	— .0007	— .0004
A. W. Blair	.3284	.9715	.0088	.3448	+ .0164	+ .0089
	.3284	.9706	.....	.3445	+ .0161	+ .0087
	.3284	[8].9209	[8].0083	[8].3270	— .0014	— .0008
	.3284	[8].9156	[8].0076	[8].3251	— .0033	— .0018
	.3284	[8].9170	.....	[8].3256	— .0028	— .0015
F. S. Shiver	.3284	.9251	.0031	.3303	+ .0019	+ .0010
	.3284	.9245	.....	.3300	+ .0016	+ .0009
C. H. Jones	.3284	.9165	.0030	.3272	— .0012	— .0006
	.3284	.9240	.....	.3299	+ .0015	+ .0008
	.3284	.9165	.....	.3272	— .0012	— .0006
	.3284	.9190	.....	.3281	— .0003	— .0002
Sulphate of potash and magnesia (K_2SO_4 c. p. + impurities):						
G. E. Patrick	.1856	[5].5132	.....	.1832	— .0024	— .0013
	.1856	[5].5145	.....	.1837	— .0019	— .0010
	.1856	[5].5144	.....	.1836	— .0020	— .0011
	.1856	[5].5148	.....	.1838	— .0018	— .0010

[1] Subsequent washing of K_2PtCl_6 with ammonium chlorid solution and alcohol gave 0.9061 g. = 0.3219 g. K_2SO_4.

[2] Subsequent washing of K_2PtCl_6 with ammonium chlorid solution and alcohol gave 0.9041 g. = 0.3212 g. K_2SO_4.

[3] Subsequent washing of K_2PtCl_6 with ammonium chlorid solution and alcohol gave 0.9061 g. = 0.3219 g. K_2SO_4.

[4] K_2PtCl_6 washed with ammonium chlorid solution.

[5] Corrected with blanks on reagents.

[6] Subsequent washing of K_2PtCl_6 with ammonium chlorid solution and alcohol gave 0.9157 g. = 0.3281 g. K_2SO_4.

[7] The barium chlorid was added largely in the dry state in preparing solution No. 2.

[8] K_2PtCl_6 washed with ammonium chlorid solution and alcohol.

TABLE II.—*Determination of potassium sulphate by the Stassfurt method*—Continued.

[Old factors.]

Substances and analysts.	K_2SO_4 taken.	K_2PtCl_6 total found.	K_2PtCl_6 from reagents.	K_2SO_4 found.	K_2SO_4 error.	K_2O error.
Sulphate of potash and magnesia (K_2SO_4 c. p. + impurities)—Cont'd.	*Gram.*	*Gram.*	*Gram.*	*Gram.*	*Gram.*	*Gram.*
A. M. Peter	0.1856	0.5112	0.0017	0.1819	—0.0037	—0.0020
	.1856	.5127	.....	.1824	— .0032	— .0017
F. B. Bomberger	.1856	.5215	.0053	.1843	— .0013	— .0007
	.1856	.5220	.....	.1845	— .0011	— .0006
	.1856	.5214	.....	.1842	— .0014	— .0008
A. W. Blair	.1856	.5467	.0064	.1929	+ .0073	+ .0039
	.1856	.5464	.....	.1928	+ .0072	+ .0039
	.1856	[1].5220	[1].0088	[1].1837	— .0019	— .0010
	.1856	[1].5203	[1].0059	[1].1831	— .0025	— .0014
	.1856	[1].5147	.....	[1].1811	— .0045	— .0024
F. S. Shiver	.1856	.5239	.0021	.1863	+ .0007	+ .0004
	.1856	.5261	.....	.1871	+ .0015	+ .0008
C. H. Jones	.1856	.5185	.0025	.1842	— .0014	— .0008
	.1856	.5170	.....	.1837	— .0019	— .0010
	.1856	.5195	.....	.1846	— .0010	— .0005
	.1856	.5190	.....	.1844	— .0012	— .0006
Kainit (K_2SO_4 c. p. + impurities):						
G. E. Patrick, solution No. 1	.1000	[2].2784	.....	.0994	— .0006	.0003
	.1000	[2].3080	.....	[3].1099	+ .0099	+ .0054
G. E. Patrick, solution No. 2	.1000	[2].2784	.....	.0994	— .0006	— .0003
	.1000	[2].2797	.....	.0998	— .0002	— .0001
	.1000	[2].2819	.....	.1006	+ .0006	+ .0003
	.1000	[2].2807	.....	.1002	+ .0002	+ .0001
	.1000	[2].2812	.....	.1004	+ .0004	+ .0002
	.1000	[2].2813	.....	.1004	+ .0004	+ .0002
A. M. Peter	.1000	.2807	.0009	.0999	— .0001	— .0001
F. B. Bomberger	.1000	.2858	.0074	.0994	— .0006	— .0003
	.1000	.2856	.....	.0993	— .0007	— .0004
	.1000	.2860	.....	.0995	— .0005	— .0003
A. W. Blair	.1000	[4].3883	.0052	[4].1370	+ .0370	+ .0200
	.1000	[5].3987	.0039	[5].1407	+ .0407	+ .0220
	.1000	[6].3830	.....	[6].1351	+ .0351	+ .0190
	.1000	[7].3996	.....	[7].1410	+ .0410	+ .0221
B. L. Hartwell	.1011	.2846	.0077	.0988	— .0023	— .0012
	.1011	.2854	.0080	.0991	— .0020	— .0011
F. S. Shiver	.1000	.2832	.0023	.1003	+ .0003	+ .0002
	.1000	.2849	.....	.1009	+ .0009	+ .0005
C. H. Jones	.1000	.2815	.0025	.0996	— .0004	— .0002
	.1000	.2803	.....	.0992	— .0008	— .0004
	.1000	.2811	.....	.0995	— .0005	— .0003
	.1000	.2810	.....	.0994	— .0006	— .0003

[1] K_2PtCl_6 washed with ammonium chlorid solution and alcohol.

[2] Corrected with blanks on reagents. 24.5 and 22 cc $BaCl_2$ solution used instead of 20 cc required.

[3] Subsequent washing of K_2PtCl_6 with ammonium acetate solution and alcohol gave 0.3052 g. = 0.1090 g. K_2SO_4.

[4] Subsequent washing of K_2PtCl_6 with ammonium chlorid solution and alcohol gave 0.2778 g. = 0.0975 g. K_2SO_4.

[5] Subsequent washing of K_2PtCl_6 with ammonium chlorid solution and alcohol gave 0.2765 g. = 0.0971 g. K_2SO_4.

[6] Subsequent washing of K_2PtCl_6 with ammonium chlorid solution and alcohol gave 0.2792 g. = 0.0981 g. K_2SO_4.

[7] Subsequent washing of K_2PtCl_6 with ammonium chlorid solution and alcohol gave 0.2746 g. = 0.0964 g. K_2SO_4.

NOTES BY THE ANALYSTS.

G. E. Patrick.—The manipulations were found somewhat troublesome, and it seems evident that this method, as it relates to sulphates, requires considerable practice to insure success. Quite a large source of error is evident in the volume of the barium sulphate. It seems to me that a correction should be made for this.

C. H. Jones.—My results by the Stassfurt method surprised me, particularly as regards their uniformity. The precipitation of the SO_3 was not so troublesome as I anticipated, although I practiced for half a day on a commercial article before taking up the official samples. In the washing of the K_2PtCl_6 I was very careful to wash by decantation, and the precipitate was in no case put on the felt until the alcohol wash, after a thorough mixing with the platinum salt, was practically colorless. This method of procedure took considerably more time than that employed in the Lindo-Gladding method, especially in the case of kainit.

A. M. Peter.—In making these determinations the instructions were closely followed, and the only departure from them was in weighing the K_2PtCl_6 on Gooch filters, washing out with hot water, and drying and weighing the Gooch again, thus eliminating any error from the presence of insoluble particles in the K_2PtCl_6. This is the practice in this laboratory, but instead of Gooch crucibles we prefer a small glass tube like a small carbon tube. Into these tubes we put a plug of glass wool and then pour on the asbestos pulp. The asbestos felt is less easily disturbed in these tubes than in Gooch crucibles, and they may be washed thoroughly with less alcohol.

A. W. Blair.—With the sulphate of potash I found some difficulty in being able to tell just when all the SO_3 was precipitated. I also found my weights entirely too high, and suspecting that the 96 per cent alcohol failed to wash out something, I used in addition the Gladding wash and 80 per cent alcohol. As you will see from my report, that made a difference.

On testing the filtrate from the Gladding wash, I found $CaSO_4$, which confirmed my suspicions.

I used from 5 to 20 cc $PtCl_4$ solution, which will account in part for the large amount of K_2PtCl_6 in reagents.

F. S. Shiver.—I am inclined to the opinion that the method gives high results. The factors used by the syndicate, which are practically our old factors, give results nearer theory than the new factors give.

F. B. Bomberger.—The instructions were closely followed in this work, except that the K_2PtCl_6, after weighing, was dissolved from the crucible with water and the latter ignited and reweighed.

The methods as submitted are not nearly so rapid as the Lindo-Gladding method, and from my experience not nearly so satisfactory.

COMMENTS BY THE REFEREE.

The method as applied to muriate of potash appears to be quite accurate, the KCl found being in some cases slightly more than was taken, in other cases slightly less. The higher results may be explained as due to impurities in the potassium platinichlorid, since, according to this method, the solution of the salt is evaporated directly with platinum solution and no attempt is made to purify the double salt. The use of the old factor compensates in a measure for such an error. The weight of KCl, which would have been obtained by the new factor, may be readily found (except in a few instances where considerably more than 0.25 gram of the salt was employed) by adding 0.0011 gram to the weights given in the table.

The results obtained in determinations of potassium sulphate in commercial sulphate of potash, sulphate of potash and magnesia, and kainit, as reported by all the analysts with the exception of Mr. Blair, are, as a rule, quite close to theory. Mr. Shiver's results invariably show a slight plus error, which would only be exaggerated by the use of the new factor, but results by Messrs. Peter, Bomberger, and Jones, in

the case of all three salts, and by Mr. Patrick in the case of sulphate of potash and sulphate of potash and magnesia, are nearer theory when calculated by the new conversion factor.

The weights of potassium sulphate found in sulphate of potash, sulphate of potash and magnesia, and kainit, as given in the table, may be readily converted into the corresponding results by the new factor by adding, respectively, 0.0016, 0.0009, and 0.0005 gram K_2SO_4.

We should not draw hasty conclusions from the results reported, as most of the analysts tried the method for the first time, and it usually requires considerable experience with a new method before the details are thoroughly mastered. This point was brought out clearly last year. Although the results of eight American chemists established the accuracy of the Lindo-Gladding method as applied to potash salts, the Stassfurt chemists, who were not accustomed to the process, reported widely discrepant results by this method.

(2) The "Optional" Method, Acidulating with Hydrochloric Acid before Precipitating the Sulphuric Acid.

The work of the association during the years 1894 and 1895 showed that in the analysis by the "optional" method of materials rich in sulphates, serious losses of potash were sustained through occlusion. With the view of avoiding this source of error, it was suggested that the barium sulphate be precipitated in an acid solution as carried out by the Stassfurt chemists. This modification of the method has been investigated during the present year by Mr. Jones.

Portions of pure potash salts were weighed out in quantities representing 0.5 gram of the four Stassfurt salts and corresponding amounts of solutions of "impurities" were added. The solution was acidified with 1 cc of concentrated HCl and the barium sulphate precipitated, as directed by the Stassfurt method. The analysis was completed as directed for the "optional" method.

Mr. Jones's results, which follow, indicate that the proposed modification has little if any advantage over the official method.

Results by modified method.

Materials.	Muriate of potash (KCl + "impurities").		Sulphate of potash 90 per cent (K_2SO_4 + "impurities").		Sulphate of potash and magnesia (K_2SO_4 + "impurities").	Kainit (K_2SO_4 + "impurities").
	Grams.	*Grams.*	*Grams.*	*Grams.*	*Grams.*	*Grams.*
KCl or K_2SO_4 taken	0.4186	0.4191	0.4600	0.4600	0.2610	0.1410
Total K_2PtCl_6 found	1.3729	1.3725	1.2825	1.2800	.7296	.3960
K_2PtCl_6 from reagents and impurities	.0085	.0085	.0090	.0090	.0080	.0053
K_2PtCl_6 corrected	1.3644	1.3640	1.2735	1.2710	.7216	.3907
KCl or K_2SO_4 found (new factors)	.4187	.4186	.4568	.4559	.2588	.1401
Error (KCl or K_2SO_4)	+ .0001	— .0005	— .0032	— .0041	— .0022	— .0009

(3) The Determination of Potash by the Lindo-Gladding Method, Evaporating Directly with Platinum Solution, without Previous Removal of Lime.

According to the original method, as described by Gladding in 1885[1] and adopted the following year by this association, the determination of potash in kainit was made by direct evaporation of a solution of the salt with platinum chlorid, the foreign salts which contaminate the potassium platinichlorid being removed by ammonium chlorid solution.

[1] United States Department of Agriculture, Division of Chemistry, Bul. 7, p. 41.

At the convention held in 1891 it was contended that the treatment with ammonium chlorid solution was not sufficient to remove calcium sulphate, and although the subject had not been submitted to the members for investigation, it was voted[1] to adopt the present official method, which depends on the precipitation of the lime with ammonium oxalate and the removal of ammonium salts by evaporation and ignition before evaporation with platinum solution.

In 1894 De Roode[2] stated that the direct evaporation method was the most satisfactory, and to support this view gave results by this method and the official Lindo-Gladding, the "optional," and the Stassfurt methods.

Vogel and Hafecke,[3] who apparently were not familiar with the original Lindo-Gladding method, refer to De Roode's article and state that the direct evaporation process, as applied to kainit, may be regarded as a combination of the Lindo-Gladding and the shorter Fresenius methods.

Last year four chemists made determinations by direct evaporation on mixtures of pure potassium sulphate and kainit "impurities." The K_2SO_4 found, as calculated by the new factor, in no case differed more than 0.0004 gram from that taken; or, in other words, the maximum error was equivalent to but 0.08 per cent K_2O in kainit. The errors by the official Lindo-Gladding method were in a number of cases considerably greater.

The following results reported by Messrs. Peter and Hartwell were obtained on K_2SO_4 c. p., mixed with kainit "impurities," and furnish further evidence that removal of lime by precipitation is unnecessary:

1. Comparison of methods.

Materials.	A. M. Peter.	B. L. Hartwell.			
	Direct evaporation.	Official method.		Direct evaporation.	
	Gram.	*Gram.*	*Gram.*	*Gram.*	*Gram.*
K_2SO_4 taken	0.1400	0.1569	0.1657	0.1553	0.1547
Total K_2PtCl_6 found	.3914	.4439	.4711	.4356	.4296
K_2PtCl_6 from impurities and reagents		.0011	.0008		
K_2PtCl_6 corrected	.3914	.4430	.4702	.4356	.4296
K_2SO_4 found (factor 0.3587)	.1404	.1589	.1687	.1562	.1541
Error, K_2SO_4	+.0004	+.0020	+.0030	+.0009	—.0006

Although the results on artificial kainit point to the accuracy of the direct evaporation process, it seemed desirable to make comparative determinations by this and the official method on a variety of samples of commercial kainit.

Mr. Peter found in a sample of kainit from the Kentucky market the same percentage of K_2O (13.35) by the two methods.

The percentage of potash in 10 samples collected in South Carolina was determined by Mr. Shiver, with the following results:

2. Comparison of methods.

Methods.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.	No. 7.	No. 8.	No. 9.	No. 10.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Official method	12.87	12.89	12.50	12.57	12.68	12.23	10.27	12.31	12.24	12.32
Direct evaporation	12.74	12.77	12.47	12.46	12.64	12.29	10.30	12.24	12.18	12.42

In the analysis of a Vermont sample Mr. Jones found 11.97 and 11.98 per cent K_2O by the official method and 11.94 and 12.02 per cent by direct evaporation.

[1] United States Department of Agriculture, Division of Chemistry, Bull. **31**, p. 158.

[2] J. Amer. Chem. Soc., **17**, 85.

[3] Land. Versuchs-Stationen, **47**, 117.

The results of the past two years, both on the mixtures of pure K_2SO_4 with kainit "impurities" and on commercial kainit, demonstrate that the direct evaporation process is fully as reliable as the official method. As it is in addition much shorter, and much more easily carried out, there is abundant reason for returning to this, the original method, which, if the two methods had been thoroughly investigated six years ago, would probably never have been abandoned.

(4) Quantities of Platinum Solution Necessary for Determination in German Potash Salts.

The quantities of platinum solution which are directed for use in the determination of potash in the Stassfurt salts by the Lindo-Gladding method (10 cc for 0.5 gram of muriate of potash and 15 cc for 0.5 gram of the other salts) are in large excess of what are needed for converting both the potash and the soda into the double salts. Five cc supply an excess of chloroplatinic acid for kainit and sulphate of potash and magnesia, and 6 cc for muriate of potash and high grade sulphate of potash. If a liberal excess be desired, 8 cc are sufficient for all of the four salts.

In 1891 the referee[1] found in a sample of double sulphate of potash 27 per cent K_2O, using 15 cc of platinum solution, and 26.98 per cent, using 8 cc; and in a sample of kainit 9.98 and 9.90 per cent, respectively.

Mr. Jones in the analysis of muriate of potash and sulphate of potash finds 8 cc sufficient in all cases.

Mr. Peter writes as follows:

In regard to the use of a smaller quantity of platinum chlorid, I will say that it is the practice of this laboratory to use only a slight excess above the amount necessary to combine with the potash. We use a solution of platinum chlorid containing about 20.6 grams platinum to the liter, 1 cc of this being equivalent to 1 per cent of K_2O, on a basis of 1 gram substance taken. We add only 1 or 2 cc more of this solution than there are supposed to be per cents of K_2O in the substance. When the liquid is nearly all evaporated, it is very easy to tell whether enough platinum has been added by removing the beaker from the water bath and allowing it to cool slightly. On inclining the beaker so that the liquid runs away from the precipitate, it is easily seen whether the liquid has the peculiar color of platinum chlorid, and if it have not, but is too light colored, more platinum solution is added. Results are entirely satisfactory and waste of platinum is avoided. Of course, this proceeding is only applicable to the Lindo-Gladding method and where the NH_4Cl wash is used.

It appears that a saving of platinum solution can be readily effected without interfering with the accuracy of the method. This is especially desirable now that the price of the metal is advancing.

Mr. Winton said, in closing his report:

"I am opposed to making any recommendations, but will say I think we ought to return to the direct evaporation process in the analysis of kainit. Every figure we have obtained in the last two years shows that this is a desirable change, and I suggest that it be made. I can hardly recommend that the Stassfurt method be adopted. The results are, many of them, very good indeed, and I think the method thoroughly reliable, but the American people are altogether too active to take up a method of that sort. I can see no advantage in adopting it, unless it prove very much more accurate. Possibly it might be well to investigate it further during the coming year. The method is a good one; the question is whether the association wishes, eventually, to adopt it or not. I will not take the responsibility of recommending that it be adopted, nor will I recommend that the referee for next year be put through the same course of sprouts that I have been.

Recommendations.

"I have made only this one recommendation, that we return to the original method for kainit. I am also tempted to recommend that we stop this extravagance in the use of platinum solution. Possibly the association prefers to investigate that subject another year. I am fully satisfied that small quantities are sufficient, and I

[1] U. S. Dept. Agr., Div. of Chem., Bull. 31, 149.

think at most of our stations smaller quantities are used. I am loth to make any recommendations on the results of the year. I therefore suggest merely that the subject be submitted for investigation the ensuing year."

Mr. Wiley conveyed to the association an invitation from the Cosmos Club to enjoy the privileges of the club, and on his own behalf invited the members to visit the Department of Agriculture and inspect the chemical laboratories. A resolution of thanks for both invitations was adopted.

The convention adjourned until 2.30 p. m.

TUESDAY—AFTERNOON SESSION.

The PRESIDENT. The order of business calls for the presentation of papers on the determination of potash. If none be presented, discussion on topics relating to the determination of potash is in order.

DISCUSSION ON POTASH.

Mr. LINDSEY. Referring to the Stassfurt method, I would like to ask the referee whether it is possible to use that method on all the multitudinous mixed fertilizers that are analyzed?

Mr. WINTON. This method is only used for the potash salts.

Mr. VON HERFF. It simply requires previous treatment with sulphuric acid, the same as in the Lindo-Gladding method. I see no reason why it should not be used in all cases.

Mr. WINTON. How would you get rid of the soluble organic matter?

Mr. VON HERFF. With sulphuric acid, the same as in the Lindo-Gladding method. It has not been tried, so far as I know, but if it work in one it is reasonable to suppose it will in the other cases.

Mr. HUSTON. Is it supposed that it is applicable in those cases where the law specifies that we must handle only potash soluble in water? It will require considerable roundabout work to use the method, as I understand it, if we are to use it in such cases, and many of us are limited to potash actually soluble in water.

Mr. VON HERFF. In what way would that interfere?

Mr. HUSTON. We have a large amount of organic matter soluble in water.

Mr. VON HERFF. What do you do in the Lindo-Gladding method in such cases? I do not see that it would complicate the method at all, except that you know you have a little more organic matter and perhaps use a drop or two more sulphuric acid.

I was not fortunate enough to hear the report read, but I believe the general experience seems to be that the Stassfurt method is somewhat difficult. Against this I may say that the main advantage claimed by the Stassfurt chemists over the Lindo Gladding method is in simplicity of execution. Perhaps with more experience any difficulties that may exist may be overcome. Every chemist must become familiar with a

method before he can use it well. It must be remembered that chemists here have long been familiar with the Lindo-Gladding method.

Mr. WINTON. With regard to results by American chemists using the Stassfurt method, they were quite good, but not quite up to——

Mr. VON HERFF (interrupting). That is a good beginning, I think.

Mr. WINTON. Very good. But the Stassfurt method certainly has no advantage over the Lindo-Gladding method so far as our results go.

Mr. VON HERFF. If both methods get good results, one would be as good as another. It is an advantage, however, to have one method adopted universally.

The PRESIDENT. I think if it comes to be understood on the other side of the water that chemists on this side get as reliable results with the Lindo Gladding method as they can with the Stassfurt method, that difficulty will be obviated. If that comes to be understood clearly, the point the gentleman has raised will be dissipated without the necessity of educating American fertilizer control chemists in a new method.

Mr. LINDSEY. It seems to me that a great deal of very careful work has been done this year in comparing these two methods. I think the point should be taken into consideration, as Mr. von Herff states that the Stassfurt method is comparatively new to our chemists, and it certainly does require some experience to get the best results with any method. On the other hand, it would seem to me that so far as they have gone the Stassfurt method does, in their hands, give quite reliable results; but it certainly is in no sense, if I understand their report, superior to the Lindo-Gladding method. The Lindo-Gladding method, it appears, can be worked more quickly than the Stassfurt, and therefore if Mr. von Herff is so desirous that we should all have one method. I think it is the sense of this association that we recommend our friends across the water to adopt the Lindo-Gladding method.

Mr. VON HERFF. When it comes to final action this is a matter of importance, and I would admit both methods, so that in case difficulties arise they can be settled on an equal basis. It is admitted by the association that the method is accurate, and therefore there is no reason to leave it out of consideration entirely. So far as simplicity is concerned, it is strongly claimed by the Stassfurt chemists that it is the quickest possible. They work it in a very short time without difficulty.

Mr. WINTON. I would like to ask our friend whether, if we adopt the Stassfurt method, the German chemists will return the compliment and adopt the Lindo-Gladding method? Possibly we can meet them halfway.

Mr. VON HERFF. I have no authority to speak for the Stassfurt chemists, and I can say nothing about that. They have no association, so far as I know. There are quite a number of chemists there and they all work this method, but I believe they would be very much

pleased to adopt the Lindo-Gladding method if there be any simplicity in it. But they say the other is very much quicker.

Mr. HILLS. Do chemists in Germany, as a rule, use the Stassfurt method?

Mr. VON HERFF. There has been issued a printed circular describing the method in detail, and I think it is used in most of the laboratories.

Mr. HILLS. For mixed fertilizers?

Mr. VON HERFF. They have not so many mixed fertilizers in Germany.

Mr. WINTON. In the matter of the simplicity of the two methods I think the only way we can arrive at a true solution of the problem is by having a competitive trial in some laboratory. But I think our friends the Stassfurt chemists are hardly acquainted with the Lindo-Gladding method as we use it. They recommend for the Stassfurt method weighed filter paper. Now, we gave that up in this country, and use instead the Gooch crucible. In their latest instructions they say the Gooch crucible may be used, but it is very evident that they are not experienced with it, and I do not think they appreciate what a very simple undertaking it is to purify K_2PtCl_6 when collected in a Gooch crucible. I think it must be very much shorter than precipitating with sulphuric acid, as they do.

Mr. JONES. In regard to the question of time in the manipulation of the two methods, I think, that so far as one sample is concerned, the time consumed in the two processes might be about the same, but with a string of a dozen or twenty samples I think the Lindo-Gladding method would beat the other 2 to 1 easily.

Mr. HILLS. I would like to ask Mr. Winton if the German chemists have tried the Lindo-Gladding method except at the one time he mentioned in this morning's talk?

Mr. WINTON. The results are given in full in last year's report. I can not say whether any of them have tried it before, but I should say from the results that they had not. The whole trouble is, I think, that they collected the salt on the weighed paper. I do not think the Lindo-Gladding method works so well when you use weighed filter paper. I do not feel, however, that it has had a fair trial on the other side of the water.

Mr. VON HERFF. I believe the question before this association is practically this: Should it be allowed to use the Stassfurt method or must the Lindo-Gladding method be used? It has been acknowledged that the Stassfurt method is an accurate one and has been used by the parties most interested in potash, therefore I do not see why it should be rejected, nor why it should be said that such and such a method is our official method and no other should be admitted, although the other is admitted to be accurate.

The PRESIDENT. If there be no further discussion, the report is referred to the committee on recommendations.

RECOMMENDATIONS.

The committee on methods of potash analyses made the following recommendations:

1. That the same method be used for determining K_2O in kainit as is now used for sulphate and double sulphates. Adopted.
2. That the referee investigate our present methods for determining K_2O as applied to wood ashes and cotton-hull ashes. Adopted.
3. That the alternate method for determining K_2O is not recommended when K_2O is present in the form of sulphate. Mr. Van Slyke suggested that the word alternate be changed to optional. Adopted.
4. That the referee for 1898 investigate methods for determining chlorin.

Mr. BARTLETT (chairman of the committee on recommendations). We found in talking the matter over that the association had no official method for determining chlorin; that several different methods were used, and that the subject was worthy of investigation.

The fourth recommendation was adopted.

The chair called for the report of the committee appointed last year to make a reply to the Stassfurt chemists on their strictures on the Lindo-Gladding method. The report was read by Mr. Winton. It was decided to offer the report for publication in the Versuchs-Stationen, the organ of the German experiment stations.[1]

Mr. SCOVELL. Will that report be printed as a part of the proceedings?

The PRESIDENT. It is presented as a part of the proceedings, and will be printed in some chemical journal.

Mr. VAN SLYKE. I think we ought to take some official action indicating the approval of the association, and I therefore move that the association approve the report as read.

Seconded and adopted.

Mr. BIGELOW. In regard to the publication of this matter, I think it was suggested last year that it would be well to have it more widely published than in our bulletin, and I move that the committee be instructed to use their own judgment as to where it shall be published, but have it printed in at least one scientific journal of large circulation.

Seconded and adopted.

Mr. WINTON. I wish to bring up a reconsideration of the motion passed this morning with regard to potash. It was moved and carried, I believe, that the optional method for the determination of potash be not recommended when the potash exists in the form of sulphate. I move that this be reconsidered. It is my purpose to refer the subject again to the committee.

The motion was seconded and carried.

Mr. Winton then moved that the question be again referred to the committee, which was adopted, and the matter declared to be so referred.

Mr. BARTLETT. The committee recommends that under the head of optional method for determination of potash, the following be

[1] The paper is published in Chemical News, vol. 77.

inserted: The Lindo-Gladding method is preferable in the presence of soluble sulphates.

Mr. WHEELER. I will say that this is merely a signboard hung up before this optional method to show that you can not get good results in the presence of sulphates.

The recommendation was adopted.

Mr. Patterson, on behalf of the committee on order of business, reported that after careful consideration it seemed best to visit the laboratories of the Department of Agriculture on Wednesday afternoon, immediately after the election of officers. Upon motion it was agreed, after some discussion, that the election of officers take place Wednesday afternoon at 2 o'clock, and that immediately thereafter the association adjourn to visit the Department of Agriculture.

Mr. WHEELER. Before the matter of phosphoric acid is taken up I would like to call the attention of the association to the movement that is on foot to secure uniform legislation on the subject of fertilizer laws. I would say that a preliminary meeting was called in New Haven at which representatives of a number of States were present, and a committee was appointed to take steps toward securing, if possible, uniform legislation in the different States. This committee met in May in New York, held a conference with a committee of the Eastern Fertilizer Association, and as a result of that meeting recommended that this association and the Association of Agricultural Colleges and Experiment Stations each appoint a committee to take such further steps as may seem desirable. The Association of Agricultural Colleges has a committee, but this association has not. It would seem appropriate that this association should join the other in the effort to secure this end, and I would therefore move that the chairman appoint a committee of five from this association to act with the committee from the other association, and that they be authorized to take such further action as may seem desirable, and report to the association at a later meeting.

Seconded.

Mr. WILEY. In supporting this motion, I would like to suggest that, as we have already appointed on that committee by the Association of Agricultural Colleges a number of gentlemen who are also members of this association, the president, in exercising his discretion in this matter, include those gentlemen on our committee, so that in that way they may represent both associations. I think at least three of the committee appointed at Minneapolis are members of this association.

Mr. WHEELER. I will say that I bring this matter up now so that those appointed upon this committee may know that we look to have them remain for a committee meeting after the close of our association, because a meeting of the committee of agricultural colleges has been called for Friday morning, and probably everything necessary to be done now can be done in a very short time.

The motion was carried. The chair announced the members of the committee later.

The report on phosphoric acid was read by Dr. McDonnell, state chemist of Maryland.

REPORT ON PHOSPHORIC ACID, IRON AND ALUMINA, AND BASIC SLAG.

By H. B. McDonnell, *Referee.*

During the latter part of January last the referee sent out a circular letter asking the cooperation of chemists in the work on phosphoric acid, and also on iron and alumina and basic slag, as directed by the association last year. Twenty-three chemists requested samples, which were sent out about March 1. A small reserve of five sets of samples was prepared, as, according to the experience of last year, this number would have been ample. This reserve, however, was soon exhausted, and I was unable to furnish samples to at least three chemists who afterwards applied.

Of the samples sent out No. 1 was the same as No. 1 of last year, having been made up at that time and kept as a reserve supply sealed in the bottles as sent out. The nature of the other samples is sufficiently indicated in the tables and instructions, except No. 8, which is described on page 56.

The results reported by the various analysts are given in the following tables: Table I gives the average of results reported on phosphoric acid; Table II, the detailed results as reported, except those by the "yellow method," which are given in Table III. Table IV gives the results on iron and aluminum, and Table V the results on basic slag.

With each set of samples was sent the following:

Directions for Work on Phosphoric Acid.

Determine total phosphoric acid in samples 1 to 6 inclusive, by—

A. The official gravimetric method.

B. The modified volumetric method. This method, which is recommended by the referee, has given excellent results in his laboratory in thousands of comparisons with the gravimetric method on "insolubles." It is as follows:

To an aliquot portion of the solution, prepared according to one of the official methods, preferably A_2, is added NH_4OH in excess, then HNO_3 in excess, then the regular molybdate solution (which should be filtered if necessary) in excess. Warm on the water bath to about 50° C. for twenty or thirty minutes, with occasional stirring; filter, wash with water at ordinary temperature until filtrate fails to show acidity when tested with litmus paper. Transfer the filter and contents to a beaker, add 20 to 40 cc of water and standard alkali sufficient to dissolve, stirring well; titrate excess of alkali with standard sulphuric or nitric acid, using phenolphthalein as indicator.

Nitric acid wash for yellow precipitate is not necessary, and nitrate solutions should be avoided, because molybdic acid is insoluble therein.

Overheating of the yellow precipitate is to be avoided, as it may cause a granular precipitate of molybdic acid which is difficult to wash out.

Good results are also obtained by heating the solution, after the addition of the molybdate, to 50° C. and allowing it to stand over night at ordinary temperature.

C. The volumetric method as given in Bulletin 46 and Circular 2, Chemical Division, United States Department of Agriculture.

D. Gladding's "yellow method" sent herewith. Mr. Gladding now recommends two final washings with alcohol in order to facilitate the drying, which is done in a glycerol bath at 105° C. (J. Am. Chem. Soc., Jan., '96, p. 23.)

Methods for the Determination of "Available" Phosphoric Acid in Basic Slag.

(Outline suggested by Mr. Wiley):

(*a*) Solubility in official ammonium citrate solution.
(*b*) Solubility in Wagner's ammonium citrate solution.
(*c*) Solubility in 1 per cent ammonium citrate solution.
(*d*) Determination of the fineness of sample.
(*e*) Determination of the per cent of sample removed by magnet.

Make determinations as follows on samples Nos. 6 and 7; the former is American, the latter European slag:

1. (*a*) Determine "available" by the regular official method.

(*b*) Determine the "available" by the regular official method, except to treat the substance with the citrate for one hour.

2. *Wagner's method.*—Five grams of the slag meal, without further grinding or sifting, are put into a half-liter flask which is filled to the mark with dilute ammonium citrate solution, the temperature of which is 17½° C. The flask is closed by means of a rubber stopper and immediately put into a revolving agitating apparatus for thirty minutes, the revolutions being made at the rate of 30 to 40 per minute, and filtered at once.

Fifty cubic centimeters of the filtrate are mixed with 100 cc of molybdate solution and heated on a water bath at 80° to 90° C. for from ten to fifteen minutes; the mixture is removed from the bath, cooled to the temperature of the room and the process continued by the usual gravimetric method.

Wagner's concentrated ammonium citrate solution contains in 1 liter 150 grams of crystallized citric acid and 23 grams of ammoniacal nitrogen. The latter is to be determined accurately by analysis by distilling off the ammonia from a measured volume of the solution (after making the solution up with approximately the proper amount of ammonia to exactly eight or nine tenths of the intended volume). The determination is made as in the Gunning method, omitting the digestion. The solution is now made up with ammonia of such strength that the total nitrogen per liter is 23 grams.

For use, two volumes of this solution are diluted with three volumes of water.

3. Treat 1 gram slag meal in an 8-ounce (250 cc) Erlenmeyer flask with 100 cc of 1 per cent citric acid solution at approximately 20° C. for one hour, shaking moderately every ten minutes. Filter through a dry filter and determine P_2O_5 in 40 or 50 cc of the filtrate by the molybdate method.

Try also the effect of treating the slag as above (under 3) for two hours instead of one hour.

4. Determine fineness on 10 grams of sample by using: (*a*) Brass sieve with circular holes ½ mm in diameter. (*b*) Sieve of bolting cloth having 4 meshes to the mm. (*c*) Sieve of bolting cloth having 10 meshes to the mm, making four separations. (Note.—After these directions were given it was found that bolting cloth with 10 meshes to the mm could not be obtained and that with 5 meshes to the mm was used instead; the latter is approximately one-tenth mm between the meshes.)

5. Determine the amount of sample removed by a magnet. Use 1 gram for the determination, spread in a thin layer on glazed paper.

METHODS FOR THE DETERMINATION OF IRON AND ALUMINA IN PHOSPHATES.

Determine iron and alumina in samples 1, 4, 5, 6, and 8 by the methods given below. Sample No. 8 is composed of pure chemicals and its composition is accurately known.

1. *Acetate method.*—Dissolve 2 to 2½ grams of the phosphate in 30 cc HCl and 10 cc HNO_3 (all iron must be in the ferric state), cool, make up to 250 cc, and take 50 cc for the determination. Add 2 drops of 1 per cent methyl orange and ammonia until alkaline. Dissolve the precipitate in very slight excess of HCl, dilute to about 300 cc, add 25 cc 20 per cent ammonium acetate solution for one hour and filter. Dissolve the precipitate in hot dilute HCl, and precipitate as before twice more, making three precipitations in all. Wash precipitates with hot water. Incinerate the precipitate in a platinum crucible, cool, add about 2 to 5 grams Na_2CO_3, or Na_2CO_3 and K_2CO_3, fuse gently from fifteen to twenty minutes, dissolve the fusion in hot water, filter and wash. Acidify the filtrate with HCl, boil off CO_2, dilute to 300 cc, and precipitate with ammonium acetate as above. Filter, wash the precipitate practically free of chlorids, ignite and weigh as $AlPO_4$, which multiplied by 0.418 equals Al_2O_3.

Dissolve the Fe_2O_3 on the filter in hot dilute HCl, precipitate with ammonia, wash free of chlorids and weigh as Fe_2O_3.

Before precipitating the iron and aluminum phosphates as above, it is probably desirable to add a little ammonium or sodium phosphate.

2. *Thiosulphate method.*—Dissolve 2 to 2½ grams of the material in 30 cc HCl in a 250 cc flask, cool, make up to mark, take 50 cc and determine iron by permanganate (Margerite's method, Fresenius Quant. Anal., p. 267, edition 1881). The HCl may be practically all removed by boiling with the H_2SO_4.

To 50 cc of the original solution add NH_4OH in slight excess, and then HCl until faintly acid. Now add 1 cc of a 1 per cent solution of KCNS, warm to about 65° C., and reduce the iron with ammonium thiosulphate, using a 2 per cent solution. (Sodium thiosulphate may be used instead.) This should be done very slowly and there should be no odor of SO_2 until after the color has entirely disappeared; the solution must remain perfectly clear. Dilute to about 300 cc, add 25 cc 50 per cent ammonium or sodium thiosulphate solution, bring to a boil and continue to boil for thirty minutes, remove from flame, let settle for five or ten minutes and filter, washing out the beaker thoroughly and twice on the filter with water at 65° C. Dissolve the $AlPO_4$ on the filter back into the beaker in which the precipitation was

made, with hot 25 per cent KOH free from Al_2O_3, washing finally several times with hot water. Make faintly acid with HCl and precipitate with ammonium thiosulphate, boiling a half hour as before; filter on a Gooch crucible, wash with warm water till free of chlorids, ignite and weigh. Multiply by 0.418 for Al_2O_3.

3. *G. Glaser's method.*—Remove metallic iron by magnet. Dissolve 2 grams in aqua regia, dilute to 250 cc and use 50 cc for analysis. Add NH_4OH until barely acid to methyl orange; add ammonium acetate (containing free acetic acid) until color turns quite yellow; heat to 70° C., let settle well; incinerate precipitate after washing with warm water, in a platinum crucible at a low heat; let cool, mix with about 2 grams $KNaCO_3$, and fuse for fifteen minutes at a gentle heat. Dissolve fusion in hot water and filter over ashless filter. Filtrate contains all Al_2O_3 and most P_2O_5. Residue on filter contains Fe_2O_3, some P_2O_5 and possibly some $CaCO_3$. Filtrate is acidified with HCl, CO_2 boiled off and $AlPO_4$ precipitated as before. After washing precipitate redissolve in warm HCl, add some pure P_2O_5 solution or $(NH_4)_2HPO_4$ (but no salt of fixed alkali), repeat precipitation as before, wash well with tepid water, incinerate and weigh as $AlPO_4$. Weight multiplied by 0.42 equals Al_2O_3. Iron residue on filter is dissolved in hot dilute HCl and washed into beaker; precipitate by NH_4OH, add sufficient c. p. citric acid to obtain a clear alkaline solution. Now ammonium sulphide is added in excess. Stir well for a short time, let settle (usually clear in ten minutes) filter over ashless filter, wash well with dilute ammonium sulphid to remove last traces of citric acid, dissolve black precipitate in dilute HCl, oxidize by HNO_3, finally precipitate with NH_4OH, wash, incinerate, and weigh as Fe_2O_3.

Where methods employed differ from directions given please give details.

It is desired that all results be reported by July 1, and in no case later than one month before the next meeting of the association, in order to give the referee ample time to tabulate the work.

H. B. McDonnell, *Referee.*

Table I.—*Average results showing content of phosphorus obtained by analysts of phosphoric acid samples.*

Analysts.	1. S. C. rock.			2. Bone.			3. Cotton-seed meal.		
	Grav.	Mod. vol.	Vol.	Grav.	Mod. vol.	Vol.	Grav.	Mod. vol.	Vol.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Stillwell and Gladding	28.16	28.20	.....	22.72	22.75	.....	2.56	2.59	.....
J. Thompson, S. C	28.09	28.20	.....	22.71	22.83	.....	2.64	2.62	.....
C. C. McDonnell, S. C	27.99	28.03	.....	22.64	22.67	.....	2.59	2.61	.....
F. S. Shiver, S. C	27.96	28.16	.....	22.60	22.58	.....	2.62	2.58	.....
M. A. Scovell, Ky	.....	[1]	28.21	.....	[2]23.62	23.29	.....	2.63	2.69
A. M. Peter, Ky	28.20	27.85	28.37	22.80	22.65	.....	2.62	2.61	.....
H. E. Curtis, Ky	28.25	28.72	28.05	22.81	23.46	23.15	2.58	2.60	2.53
Ora K. Knight, Me	28.08	27.46	.....	23.22	22.70	.....	2.65	2.70	.....
C. B. Williams, N. C	27.37	27.51	27.80	22.36	22.34	22.78	2.57	2.61	2.62
C. D. Harris, N. C	27.63	.....	.....	22.39	.....	.....	2.54	.....	.....
E. G. Runyan, D. C	28.21	27.75	27.60	22.99	22.85	22.60	2.56	2.52	2.54
C. H. Jones, Vt	28.18	28.25	.....	23.08	23.05	.....	2.65	2.49	.....
B. O. White, Vt	28.22	.....	.....	23.12	.....	.....	2.56	.....	.....
W. L. Mitchell, Conn	28.13	28.29	.....	22.52	22.64	.....	2.53	2.55	.....
W. H. Andrews, N. Y	27.70	28.25	.....	22.78	22.86	.....	2.57	2.68	.....
A. D. Cook, N. Y	27.59	27.51	.....	23.07	23.06	.....	2.50	2.56	.....
C. W. Norris, Pa	28.31	[2]29.19	.....	22.86	[2]24.12	.....	2.62	2.78	.....
F. P. Veitch, Md	28.01	28.02	.....	22.61	22.99	23.17	2.52	2.67	.....
W. Skinner, Md	28.02	27.92	.....	23.04	23.02	.....	2.66	2.68	.....
Difference between highest and lowest	.94	1.73	.77	.76	1.78	.69	.16	.29	.16
Greatest difference from average	.63	1.18	.41	.43	1.24	.29	.07	.13	.06
Number averaged	18	15	5	18	15	5	18	17	4
Average	28.00	28.01	28.01	22.79	22.83	23.00	2.59	2.62	2.59

[1] Too discordant for average.

[2] Omitted from average.

TABLE I.—*Average results showing content of phosphorus, etc.*—Continued.

Analysts.	4. Fla. phosphate.			5. Ala. phosphate.			6. Pottstown slag.		
	Grav.	Mod. vol.	Vol.	Grav.	Mod. vol.	Vol.	Grav.	Mod. vol.	Vol.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Stillwell and Gladding	30.16	30.10	.....	.....	42.00	.....	16.70	16.60	.....
J. Thompson, S. C	30.15	30.33	.....	42.23	42.37	.....	16.28	16.27	.....
C. C. McDonnell, S. C	30.17	30.18	.....	42.24	42.74	.....	16.26	16.34	.....
F. S. Shiver, S. C	30.16	30.25	.....	42.35	42.10	.....	15.91	16.03	.....
M. A. Scovell, Ky	.....	[1]31.62	30.70	.....	([2])	43.15	.....	16.61	.....
A. M. Peter, Ky	30.04	30.15	30.60	42.15	42.50	43	16.16	16.75	16.24
H. E. Curtis, Ky	30.10	30.92	30.45	43.14	43.92	43.40	16.51	16.48	16.05
Ora K. Knight, Me	30.04	29.92	.....	42.53	42.30	.....	15.97	15.47	.....
C. B. Williams, N. C	29.27	29.14	29.90	41.40	41.37	41.59	15.69	15.70	16.26
C. D. Harris, N. C	29.31	.....	.....	41.23	.....	.....	15.67	.....	.....
E. G. Runyan, D. C	30.22	30.05	29.90	42.26	42.23	41.90	16.08	15.75	15.80
C. H. Jones, Vt	30.30	30.28	.....	42.56	42.54	.....	16.40	16.48	.....
B. O. White, Vt	30.40	.....	.....	42.64	.....	.....	16.40	.....	.....
W. L. Mitchell, Conn	30.58	30.39	.....	42.91	42.62	.....	16.30	16.14	.....
W. H. Andrews, N. Y	30.21	30.64	.....	42.53	42.69	.....	16.18	16.11	.....
A. D. Cook, N. Y	30.36	30.47	.....	42.36	42.41	.....	16.29	16.08	.....
C. W. Norris, Pa	30.24	[1]31.45	.....	42.84	[1]44.26	.....	16.69	[1]16.89	.....
F. P. Veitch, Md	29.97	29.92	.....	42.62	42.23	.....	16.39	16.56	.....
W. Skinner, Md	30.13	30.06	.....	42.37	41.97	.....	16.63	16.54	.....
Difference between highest and lowest	1.31	2.48	.80	1.91	2.89	1.81	1.03	1.42	.46
Greatest difference from average	.48	1.14	.39	.77	1.03	.79	.45	.66	.17
Number averaged	18	15	5	17	15	5	18	16	4
Average	30.10	30.19	30.31	42.37	42.40	42.61	16.25	16.23	16.09

[1] Omitted from average.

[2] Too discordant for average.

TABLE II.—*Detailed results on phosphoric acid as reported by analysts.*

Analysts.	1. S. C. rock.			2. Bone.			3. Cotton-seed meal.			(1)
	Grav.	Mod. vol.	Vol.	Grav.	Mod. vol.	Vol.	Grav.	Mod. vol.	Vol.	
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	
Stillwell and Gladding, N. Y	28.16	28.20	.....	22.72	22.75	.....	2.56	2.59	.....	
John Thompson, S. C	28.09	28.20	.....	22.71	22.83	.....	2.64	2.62	.....	A_5
C. C. McDonnell, S. C	27.99	28.03	.....	22.64	22.67	.....	2.59	2.61	.....	A_5
	28.05	.....	.....	22.68	.....	.....	2.59	.....	.....	([3])
F. S. Shiver, S. C	27.96	28.16	.....	22.60	22.58	.....	2.62	2.58	.....	([3])
M. A. Scovell, Ky	.....	[4]28.32	[4]28.40	.....	23.62	23.40	.....	2.64	2.70	A_1
	.....	[4]29.61	[4]28.15	.....	.....	23.60	.....	2.62	2.68	A_1
	.....	.....	[4]28.15	.....	.....	22.85	.....	.....	.....	A_1
	.....	.....	[4]28.10	.....	.....	22.95	.....	.....	.....	A_1
	.....	.....	[4]28.25	.....	.....	23.67	.....	.....	.....	A_1
	.....	.....	[4]27.90	.....	.....	[5]21.75	.....	.....	.....	
A. M. Peter, Ky	[5]28.20	[5]27.85	[6]28.10	[7]22.80	[7]22.65	.....	[7]*a*2.62	[7]*a*2.61	.....	

[1] See Bulletin No. 46, Division of Chemistry, page 12.

[2] Dissolved in H_2SO_4 and KNO_3.

[3] Volumetrics stood over night.

[4] Dissolved by A_7.

[5] Dissolved by A_5.

[6] HA and HNO_3.

[7] HNO_3.

[7*a*] Mg $(NO_3)_2$.

TABLE II.—*Detailed results on phosphoric acid as reported by analysts*—Continued.

Analysts.	1. S. C. rock.			2. Bone.			3. Cotton-seed meal.			(1)
	Grav.	Mod. vol.	Vol.	Grav.	Mod. vol.	Vol.	Grav.	Mod. vol.	Vol.	
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	
A. M. Peter, Ky	.....	[2] 29.20	[3] 28.40	.....	[2] 23.30	.....	.....	.....	.....	
	.....	.....	[3] 28.60	.....	.....	.....	.....	.....	.....	
H. E. Curtis, Ky	28.25	28.72	28.05	22.80	23.46	23.15	2.58	2.60	2.53	(4)
	28.25	.....	.....	22.82	.....	.....	2.57	.....	.....	
Ora K. Knight, Me	[5] 28.08	27.46	.....	[5] 23.22	22.72	.....	[5] 2.65	2.62	.....	
	[6] 28.15	[7] 27.38	.....	[6] 23.06	22.68	.....	[6] 2.56	2.79	.....	
	[8] 28.18	.....	.....	[8] 23.30	[7] 22.68	.....	[8] 2.72	.....	.....	
C. B. Williams, N. C	27.34	27.51	27.75	22.32	22.29	22.73	2.58	2.57	2.55	
	27.40	27.50	27.85	22.40	22.38	22.83	2.55	2.64	2.68	
C. D. Harris, N. C	27.67	.....	.....	22.29	.....	.....	2.52	.....	.....	
	27.58	.....	.....	22.48	.....	.....	2.56	.....	.....	
E. G. Runyan, D. C	28.21	27.75	27.60	22.99	22.85	22.60	2.56	2.52	2.54	
W. L. Mitchell, Conn	28.12	28.23	.....	22.43	22.68	.....	2.51	2.53	.....	
	28.00	28.36	.....	22.61	22.61	.....	2.56	2.57	.....	(3)
	28.20	.....	.....	.....	.....	.....	.....	.....	.....	
	28.20	.....	.....	.....	.....	.....	.....	.....	.....	
C. H. Jones, Vt	[10] 28.18	[11] 28.30	.....	[10] 23.08	[11] 23.00	.....	[10] 2.65	[12] 2.49	.....	
	.....	[13] 28.25	.....	.....	[13] 23.10	.....	.....	.....	.....	
	.....	[12] 28.20	.....	.....	[12] 23.05	.....	.....	.....	.....	
B. O. White, Vt	[10] 28.22	.....	.....	[10] 23.12	.....	.....	[10] 2.56	.....	.....	
W. H. Andrews, N. Y.	27.60	28.20	200 cc H_2O	22.80	22.85	.....	2.62	2.69	.....	
	27.74	28.22		22.76	22.86	.....	2.55	2.68	.....	
	27.75	28.20		.....	22.87	.....	2.55	2.68	.....	
	.....	28.30	300 cc H_2O	.....	.....	.....	.....	.....	.....	
	.....	28.35		.....	.....	.....	.....	.....	.....	
A. D. Cook, N. Y	27.50	27.41	.....	23.08	23.08	.....	2.50	2.54	.....	
	27.68	27.60	.....	23.06	23.04	.....	2.50	2.58	.....	
C. W. Norris, Pa	28.33	29.30	28.46	22.76	23.83	23.64	2.60	2.75	2.74	
	28.47	29.28	28.95	23.02	24.41	23.40	2.60	2.81	2.79	
	28.29	28.99	28.85	22.74	.....	24.26	2.69	.....	2.75	
	28.14	.....	.....	22.75	.....	.....	2.63	.....	.....	
	.....	.....	.....	23.02	.....	.....	.....	.....	.....	
F. P. Veitch, Md	28.01	27.90	.....	22.60	22.90	23.17	2.57	2.68	.....	
	28.01	27.95	.....	22.68	22.85	.....	2.46	2.65	.....	
	.....	28.10	.....	22.68	[14] 23.07	.....	.....	.....	.....	
	.....	[14] 28.00	.....	22.89	[14] 23.15	.....	.....	.....	.....	
	.....	[14] 28.15	.....	22.52	[14] 23.00	.....	.....	.....	.....	
W. Skinner, Md	27.99	27.88	.....	23.03	23.01	.....	2.62	2.68	.....	
	28.06	27.96	.....	23.02	23.09	.....	2.70	2.68	.....	
	28.02	27.91	.....	23.10	22.96	.....	.....	.....	.....	

[1] See Bulletin No. 46, Division of Chemistry, page 12.

[2] Keldahl solution.

[3] HA and HNO_3.

[4] Dissolved in H_2SO_4—A_4.

[5] 0.5 gram.

[6] 0.5 gram, not neutralized.

[7] Not neutralized.

[8] 0.2 gram, not neutralized.

[9] Special molybdate solution. J. Am. Chem. Soc., Mar., 1896.

[10] A_4.

[11] A_5.

[12] A_2.

[13] A_5 stood overnight.

Method of solution, Bull. 46, page 12.

[14] Stood twelve hours: $Mg(NO_3)_2$, method on No. 3.

TABLE II.—*Detailed results on phosphoric acid as reported by analysts*—Continued.

Analysts.	4. Fla. phosphate.			5. Ala. phosphate.			6. Pottstown slag.			(1)
	Grav.	Mod. vol.	Vol.	Grav.	Mod. vol.	Vol.	Grav.	Mod. vol.	Vol.	
Stillwell and Gladding, N. Y.	*Per ct.* 30.16	*Per ct.* 30.10	*Per ct.*	*Per ct.*	*Per ct.* 42.00	*Per ct.*	*Per ct.* 16.70	*Per ct.* 16.60	*Per ct.*	
John Thompson, S. C.	30.15	30.33	.....	42.23	42.37	.....	16.28	16.27	.....	A_5
C. C. McDonnell, S. C.	30.17	30.18	.....	42.24	27.74	.....	16.26	16.34	.....	A_5
	30.15	.....	.....	42.35	.....	.....	16.28	.....	.....	(2)
F. S. Shiver, S. C.	30.16	30.25	.....	42.35	42.10	.....	15.91	16.03	.....	(3)
	.....	31.68	31.30	.....	45.69	43.90	.....	16.48	16.60	
	.....	31.55	31.05	.....	44.08	42.85	.....	16.74	16.50	
	.....	.....	30.10	.....	.....	42.85	.....	.....	15.95	A_4
	.....	.....	30.75	.....	.....	43.30	.....	.....	15.80	A_4
	.....	.....	29.95	.....	.....	42.85	.....	.....	16.50	
	.....	.....	31.05	.....	.....	.....	.....	.....	.....	
	.....	.....	[4]29.45	.....	.....	[4]42.30	.....	.....	[4]15.85	
A. M. Peter, Ky.	[5]30.04	[5]30.15	[6]30.45	[5]42.14	[5]42.50	[6]42.90	[5]16.14	[5]16.45	[6]16.25	
	.....	[7]31.70	[6]30.75	[5]42.16	[7]45.60	[6]43.10	[5]16.17	[7]17.00	[6]16.23	
	.....	.....	.....	.....	[7]43.20	.....	.....	.....	.....	
	.....	.....	.....	.....	[7]43.60	.....	.....	.....	.....	
	.....	.....	.....	.....	[7]44.00	.....	.....	.....	.....	
H. E. Curtis, Ky.	30.11	30.92	30.45	43.54	43.92	43.40	16.66	16.48	16.05	(8)
	30.09	.....	.....	43.24	.....	.....	16.63	.....	.....	(8)
	.....	.....	.....	43.08	.....	.....	.....	.....	.....	
	.....	.....	.....	[9]42.68	.....	.....	[9]16.25	.....	.....	
Ora K. Knight, Me.	[10]29.99	29.92	.....	[10]42.44	42.28	.....	[10]15.97	15.40	.....	
	[10]30.08	29.92	.....	[10]42.62	42.32	.....	[11]16.03	15.53	.....	
	.....	[12]29.88	.....	.....	[12]42.03	.....	[13]15.99	[12]15.48	.....	
C. B. Williams, N. C.	29.21	29.08	29.86	41.31	41.19	41.59	15.76	15.52	16.31	
	29.33	29.20	29.93	41.48	41.54	.....	15.61	15.87	16.21	
C. D. Harris, N. C.	29.37	.....	.....	41.13	.....	.....	15.66	.....	.....	
	29.24	.....	.....	41.33	.....	.....	15.67	.....	.....	
E. G. Runyan, D. C.	30.22	30.05	29.90	42.26	42.23	41.90	16.08	15.75	15.80	
W. L. Mitchell, Conn.	30.59	30.29	.....	42.79	42.92	.....	16.26	16.14	.....	
	30.55	30.24	.....	42.60	42.99	.....	16.36	16.14	.....	
	30.60	30.39	.....	.....	42.93	.....	16.29	.....	.....	(14)
	.....	30.59	.....	.....	42.80	.....	.....	.....	.....	(14)
	.....	30.40	.....	.....	.....	.....	.....	.....	.....	
	[11]29.94	.....	.....	[11]42.88	.....	.....	.....	.....	.....	
	[13]30.01	.....	.....	[13]42.64	.....	.....	.....	.....	.....	
	[13]29.91	.....	.....	.....	.....	.....	.....	.....	.....	
C. H. Jones, Vt.	[15]30.30	[16]30.20	.....	[15]42.51	[16]42.53	.....	[15]16.36	[16]16.45	.....	
	.....	[17]30.25	.....	[8]42.60	[17]42.50	.....	[8]16.44	[17]16.45	.....	
	.....	[8]30.40	.....	.....	[8]42.60	.....	.....	[8]16.55	.....	
B. O. White, Vt.	[15]30.40	.....	.....	[15]42.64	.....	.....	[15]16.40	.....	.....	
W. H. Andrews, N. Y.	30.16	30.60	200 cc H_2O	42.50	42.55	200 cc	[18]16.39	16.10	.....	
	30.18	30.65		42.76	42.72		[19]16.08	16.12	.....	
	30.28	30.70		42.32	42.80		[19]16.08	.....	.....	

[1] See Bulletin No. 46, Division of Chemistry, page 12.
[2] Dissolved in H_2SO_4 and KNO_3.
[3] Volumetrics stood over night.
[4] Dissolved by A_7.
[5] Dissolved by A_5.
[6] HA and HNO_3.
[7] Keldahl solution.
[8] A_2. Method of solution, Bul l. 46, page 12.
[9] H_2SO_4.
[10] 0.5 gram.
[11] 0.5 gram, not neutralized.
[12] Not neutralized.
[13] 0.2 gram, not neutralized.
[14] Special molybdate solution. J. Am. Chem. Soc., Mar., 1896.
[15] A_4.
[16] A_5.
[17] A_5 stood overnight.
} Method of solution, Bull. 46, page 12.
[18] Precipitated once with molybdate.
[19] Precipitated twice.

TABLE II.—*Detailed results on phosphoric acid as reported by analysts*—Continued.

Analysts.	4. Fla. phosphate.			5. Ala. phosphate.			6. Pottstown slag.			(1)
	Grav.	Mod. vol.	Vol.	Grav.	Mod. vol.	Vol.	Grav.	Mod. vol.	Vol.	
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	
W. H. Andrews, N. Y.	.....	30.65	300 cc	.....	42.68	300 cc	.....	.....	.....	
	.....	30.60								
A. D. Cook, N. Y.	30.28	30.38	.....	42.40	42.50	.....	16.24	16.08	.....	
	30.44	30.55	.....	42.32	42.32	.....	16.34	16.08	.....	
C. W. Norris, Pa.	30.30	31.45	31.12	43.02	44.39	44.04	[2]16.81	16.79	16.79	
	30.20	31.65	31.16	42.91	44.05	43.81	[2]16.66	16.98	16.69	
	30.19	31.26	31.12	42.64	44.34	44.19	[2]16.57	.....	16.50	
	30.19	.....	.....	42.98	.....	.....	[2]16.65	.....	.....	
	30.30	.....	.....	42.66	.....	.....	[2]16.76	.....	.....	
	30.25	.....	.....	.....	.....	.....	[2]16.67	.....	.....	
F. P. Veitch, Md.	29.91	29.95	.....	42.65	42.10	.....	16.43	16.60	.....	
	30.03	[3]30.00	.....	42.57	42.19	.....	16.27	16.65	.....	
	.....	[3]29.80	.....	42.66	[3]42.32	.....	16.27	[23]16.50	.....	
	.....	.....	.....	.....	[3]42.32	.....	16.52	[23]16.55	.....	
	.....	.....	.....	.....	.....	.....	16.20	[23]16.49	.....	
	.....	.....	.....	.....	.....	.....	16.65	.....	.....	
W. Skinner, Md.	30.09	30.00	.....	42.28	42.00	.....	16.62	16.60	.....	
	30.15	30.10	.....	42.46	41.95	.....	16.65	16.55	.....	
	30.18	30.02	.....	42.36	41.95	.....	16.62	16.48	.....	

[1] See Bulletin No. 46, Division of Chemistry, page 12.

[2] Used diluted acid in dissolving.

[3] Stood twelve hours; $Mg(NO_3)_2$, Method on No. 3.

TABLE III.—*Phosphoric acid by "yellow method."*

Analysts.	Sample.					
	1.	2.	3.	4.	5.	6.
Stillwell and Gladding	28.10	22.76	2.56	30.15	42.20	16.60
	28.20	22.76	2.58	30.15	42.11	16.70
Average	28.15	22.76	2.57	30.15	42.16	16.65
E. G. Runyan, U. S. Dept. of Agr.	28.28	23.01	2.61	30.27	42.41	15.90
O. W. Knight, Me. (dried in water oven)	28.24	23.30	2.44	29.95	42.44	16.11
	28.20	23.30	2.59	.....	42.46	16.00
Average	28.22	23.30	2.52	29.95	42.45	16.06
C. W. Norris, Pa. (Dried in ordinary air bath at 105–112° C.)	28.02	22.93	2.67	30.13	42.64	16.22
	28.42	22.97	2.74	30.51	43.27	16.69
	27.94	23.15	2.53	30.95	42.14	17.18
	.....	.....	2.47	29.82	.....	.....
Average	28.13	23.02	2.60	30.35	42.68	16.70

NOTES OF ANALYSTS.

T. S. Gladding.—I find the same results by all three methods for P_2O_5. The gravimetric is least reliable. The volumetric is all right when solutions are accurate. My gravimetric "yellow method" I find most *certain* in the hands of our assistants. Its simplicity and gravimetric features are its strong points.

A. M. Peter, Kentucky.—We have had a great deal of trouble in working with the volumetric methods, and have not succeeded in getting good results, especially on

the sulphuric-acid solutions. You will notice when you compare my results with those of Mr. Curtis that there is also a considerable discrepancy between his figures and mine on samples Nos. 5 and 6 by the gravimetric method. It would appear that the results obtained from sulphuric-acid solutions by the official gravimetric method are considerably too high and that the duplicates do not agree where there is much iron and alumina.

H. E. Curtis, Kentucky.—Quite a number of determinations have been made on hydrochloric and nitric acid solutions on these same samples, but the methods varied slightly from both yours and Mr. Kilgore's, and as yet I have not been able to get duplicate results to agree. In some sets they would agree and the same work would be duplicated, and the duplicates would not agree, neither would they agree with the former results.

O. W. Knight, Maine.—In the Gladding method I used Gooch crucibles. After four hours drying in the water oven, a constant weight was reached by the precipitate. This method gives good results and seems likely to prove of value in some cases.

In the volumetric method there is great difficulty in determining the exact moment when the color change takes place. Some of the precipitate begins to dissolve in many cases before it is thoroughly washed.

C. B. Williams, North Carolina.—In the gravimetric method twenty washings were required to rid the white precipitate of chlorids.

In washing the yellow precipitate free from nitric acid, in determining phosphoric acid by your modification of the volumetric method, it was found that it required from twelve to fifteen washings, while by Kilgore's method six were sufficient.

In the manipulation of your modification the following objections are encountered: First, in consequence of the low temperature of precipitation the yellow precipitate obtained is in such a finely divided condition that it tends to run through the filter paper; second, the washing of the ammonium phosphomolybdate is so slow that there is great danger of decomposition of the yellow precipitate taking place; third, the amount of filter paper is necessarily so large in the titrating beaker that it interferes with the detection of the exact neutral point in titration. In Kilgore's method none of these objections has to be contended with. In all volumetric determinations 20 cc solutions, corresponding to 0.2 gram substance, were used. All results are from the same solution.[1]

C. H. Jones, Vermont.—I am of the opinion that if a volumetric method is to be adopted officially your modification is more nearly correct than the one printed (in Bul. 46).

Winton (Mitchell), Connecticut.—* * * Used modified molybdic solution as described in the Journal of the American Chemical Society, May, 1896.

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[1] Note by referee.—I am unable to reconcile all of the statements above with the results reported by Mr. Williams. The averages of his results, which are from closely agreeing duplicates, are as follows:

Sample.	Gravimetric.	Referee's volumetric.	Kilgore's volumetric.
No. 1	27.37	27.51	27.80
No. 2	22.33	22.34	22.78
No. 3	2.57	2.61	2.62
No. 4	29.27	29.14	29.90
No. 5	41.40	41.37	41.59
No. 6	15.69	15.70	16.26

In every case the referee's method gives results very close to the gravimetric, while in each case higher results are obtained by Kilgore's method. I do not think that the latter precipitates were sufficiently washed.

We do not think that the ammonium nitrate contained in the ordinary molybdic solution is sufficient when no more is added or formed by neutralization. As we ignite fertilizers containing organic matter with magnesium nitrate and dissolve in HCl we get no nitrate by neutralization of the solvent. We have our choice of four plans of procedure if we wish more ammonium nitrate than moderate excess of molybdic solution will furnish: (1) Addition of HNO_3 and NH_4OH; (2) addition of solid or dissolved NH_4NO_3; (3) addition of large excess of molybdic (the old way); (4) use of molybdic solution containing the extra NH_4NO_3. The latter plan seems to me preferable from point of convenience. * * *

The few determinations I have made convince me that the volumetric method, both as Kilgore left it and in the form you use it, is, when carefully carried out, thoroughly accurate. The following experiments may be of interest to you:

SERIES I.—Added HNO_3 and neutralized with NH_4OH.

Determination.	Volumetric.	Gravimetric.
	Per cent.	*Per cent.*
Total phosphoric acid in acid phosphate	17.40	17.28
Total phosphoric acid in tankage	14.83	15.05
Total phosphoric acid in dry ground fish	7.39	7.28
Total phosphoric acid in dissolved bone black	17.26	17.24
Total phosphoric acid in air-dry carnation blossoms	.65	.63
Total phosphoric acid in mixed fertilizer	6.44	6.33

SERIES II.—Added solid NH_4NO_3.

Determination.	Volumetric.	Gravimetric.
	Per cent.	*Per cent.*
Total phosphoric acid in dry ground fish	6.89	6.87
Total phosphoric acid in mixed fertilizer	11.03	10.87
Total phosphoric acid in mixed fertilizer	12.39	12.22
Total phosphoric acid in mixed fertilizer	12.04	11.88
Total phosphoric acid in mixed fertilizer	11.08	10.98
Total phosphoric acid in mixed fertilizer	10.57	10.43
Total phosphoric acid in mixed fertilizer	2.07	1.95
Soluble phosphoric acid in mixed fertilizer	7.04	6.98
Soluble phosphoric acid in dissolved bone black	12.04	11.89

SERIES III.—Using molybdic solution containing extra NH_4NO_3.

Determination.	Volumetric.	Gravimetric.
	Per cent.	*Per cent.*
Soluble phosphoric acid in mixed fertilizer	2.60	2.46
Soluble phosphoric acid in mixed fertilizer	2.40	2.32
Soluble phosphoric acid in mixed fertilizer	4.75	4.61
Soluble phosphoric acid in mixed fertilizer	9.03	8.94
Soluble phosphoric acid in mixed fertilizer	4.75	4.65
Soluble phosphoric acid in mixed fertilizer	6.69	6.51

F. P. Veitch, Maryland.—Washings in the volumetric method were from twelve to twenty-four times, amounting to from 150 cc to 250 cc on each sample, the larger amount being required by the heaviest precipitates.

I have found it safer to use 1 cc of 1 per cent phenolphthalein, as with a smaller amount the color fades too soon, giving slightly high results.

OBSERVATIONS BY REFEREE.

It is worthy of note that outside of the referee's laboratory only three of the chemists taking part in this work did so last year. Taking this into account, and also the nature of the samples, I consider this work very good indeed. There are very few wild results by the gravimetric method, and only a few by the modified volumetric method, and those are confined to three or four analysts. The average results by the latter give a better showing than those by the published method. To be sure, the variations are less by the latter method, but this loses weight when we remember that three times as many chemists tried the new modification.

I wish to again call the attention of the association to the optional method for preparing ammonium citrate. At the Chicago meeting I called attention to this method. In the first place, it is supposed that on mixing two solutions which react on each other, causing a precipitate, the filtrate will still be neutral, provided the original solutions are neutral. Although this is the rule, there are some well-known exceptions. Let us examine the method. "Dilute to nearly sp. gr. 1.09. Prepare calcium chlorid solution; make the latter neutral. To 10 cc citrate add 50 cc calcium chlorid solution. (This dilutes the citrate to one-sixth.) Filter and *dilute* the filtrate with an equal volume of water," making the acidity or alkalinity one-twelfth of the original citrate. Anything can be made neutral to the ordinary tests by the process of dilution alone, if it be carried far enough.

In the referee's laboratory the best method yet found for preparing citrate of ammonia is that of De Roode. The citric acid is put into a large evaporating dish or enameled iron pan and covered with water; ammonia is added from time to time, regulating the amount so that the hea tgenerated will not be extreme, till it is in decided excess; the dish or pan is then allowed to stand, uncovered, for about two days, when the solution will be found to be neutral. It is then diluted to the proper specific gravity.

RECOMMENDATIONS.

1. That the volumetric method, in its simplified form, be adopted as an alternate method.

2. That the De Roode method for the preparation of ammonium citrate be tested by the referee the coming year with the view of adopting it instead of the present "optional" method.

DETERMINATION OF IRON AND ALUMINA. (TABLE IV.)

Sample No. 8 was made up by using chemically pure salts, and contained one part of ferrous ammonium sulphate, one part of aluminum sulphate, and two parts of tricalcium phosphate. It contained, as nearly as could be determined from analysis of the ingredients, from 4 to 4.05 per cent of Al_2O_3 and from 5.10 to 5.15 per cent of Fe_2O_3. Analysis showed the following composition of the ingredients:

	Per cent.
Fe_2O_3 in iron ammonium sulphate, by precipitation with ammonia	20.40
	20.46
By titration with permanganate	20.26
	20.26

Average, 20.36 per cent; theory, 20.40 per cent.

	Per cent.	Per cent.
Al_2O_3 in aluminum sulphate	15.95 (Veitch);	15.94 (Bomberger).
	15.66	15.93

Average, 15.95 per cent.

The calcium phosphate used gave 0.36 per cent of iron and alumina phosphates by the acetate method. This result is thought to be considerably too high, but the determination was not repeated. The solution gave a slight reaction for iron by qualitative test.

TABLE IV.—*Results on iron and alumina.*

Analysts.	1. S. C. rock.			4. Fla. phosphate.			5. Ala. phosphate.		
	Acetate.	Thio.	Glaser.	Acetate.	Thio.	Glaser.	Acetate.	Thio.	Glaser.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
C. Glaser, Balto	.....	1.14	1.90	.....	2.23	2.26	.....	29.57	29.63
	.....	0.77	1.75	.....	.....	.....	.....	.....	29.63
	.....	1.11	1.84	.....	.....	.....	.....	.....	.....
F. S. Shiver, S. C.[2]	2.07	.....	.....	2.35	.....	.....	29.44	.....	.....
	[3]1.95	.....	.....	[3]2.37	.....	.....	29.48	.....	.....
E. G. Runyan, U. S. Dept. of Agr	1.86	1.17	1.15	2.39	1.40	0.80	27.90	27.58	24.70
F. P. Veitch, Md	1.70	1.50	.....	1.94	1.75	.....	27.12	27.81	.....
		By K MnO_4.			By K MnO_4.			By K MnO_4.	
C. Glaser	.....	[4]3.12	3.08	.....	[4]1.45	1.47	.....	10.85	10.75
	.....	[5]3.19	.....	.....	.....	.....	.....	.....	10.80
F. S. Shiver	.....	2.51	.....	.....	1.43	.....	.....	10.46	.....
E. G. Runyan	2.87	3.30	3.10	1.50	1.42	2.16	11.60	11.32	12.14
F. P. Veitch	3.06	2.94	.....	1.45	1.58	.....	11.26	10.81	.....

Analysts.	6. Pottstown slag.			8. $Al_2(SO_4)_3$: Fe *Am* SO_4: $Ca_3(PO_4)_2$.				
	Acetate.	Thio.	Glaser.	Acetate.	Thio.	Glaser.	Glad. potash.	
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	
Stillwell & Gladding, N. Y.[1]	.....	.....	.....	.....	5.07	.....	4.05	
	.....	.....	.....	.....	5.11	.....	4.01	
	.....	.....	.....	.....	3.60	.....	4.01	
C. Glaser, Balto	.....	[2]2.71	0.08	.....	4.09	4.10	.....	
	.....	.....	0.16	.....	4.03	3.96	.....	
F. S. Shiver, S. C.[2]	0.30	.....	.....	3.77	.....	.....	.....	Al_2O_3.
	0.18	.....	.....	3.79	.....	.....	.....	
	.....	.....	.....	[2]3.87	.....	.....	.....	
E. G. Runyan, U. S. Dept. of Agr	0.50	0.78	0.27	3.72	3.22	3.28	.....	
F. P. Veitch, Md	0.75	0.59	.....	3.47	3.95	.....	.....	
		By K MnO_4.			By K MnO_4.			
Stillwell & Gladding, N. Y.	.....	.....	.....	.....	.....	.....	4.92	
	.....	.....	.....	.....	.....	.....	4.93	
C. Glaser	.....	21.42	20.29	.....	5.69	5.35	.....	
	.....	.....	21.79	.....	.....	5.40	.....	Fe_2O_3.
F. S. Shiver	.....	22.38	.....	.....	5.13	.....	.....	
E. G. Runyan	23.10	23.80	22.30	5.60	5.78	6.20	.....	
F. P. Veitch	22.55	22.00	.....	5.14	5.17	.....	.....	

[1] Mostly SiO_2.
[2] Results by subtracting Fe_2O_3 from iron and alumina.
[3] Jones—Acetate method.
[4] Precipitated in 50 cc in presence of citric acid by Am. sulphid.
[5] By nitroso β naphthol.

The preliminary work on methods was done by Mr. F. P. Veitch, who got very promising results in some preliminary work by using sodium thiosulphate for the determination of alumina. Mr. Veitch got some suggestions from Blair's Analysis of Iron and Steel and one or two other places, and thought the reagent could be used for the present purpose. In later work the method did not prove all that could be desired. Some of the preliminary work done is as follows:

Prepared three solutions, 1 liter of which contained the substances and amounts of same as given below.

Composition of samples.

No. 1.	No. 2.	No. 3.
16 gms. $Ca_3(PO_4)_2$.	14 gms. $Ca_3(PO_4)_2$.	1.500 gms. $CaCl_2$ (fused).
0.4 gm. $MgCl_2$.	0.2 gm. $MgCl_2$.	4.000 gms. Na_2HPO_4.
0.2 gm. NaCl.	0.2 gm. NaCl.	0.500 gm. $MgCl_2$.
0.2 gm. KCl.	0.8426 gm. Fe.	0.250 gm. Fe (wire).
0.2902 gm. Fe.	3.0082 gms. $Al_2(SO_4)_3.18H_2O$.	2 gms. $Al_2(SO_4)_3.18H_2O$.
7.0050 gms. $Al_2(SO_4)_3.18H_2O$.		

Fifty cubic centimeters of these solutions were used in each case for analysis. The acetate method was carried out according to instructions given, except as noted.

Caustic-potash method.

Precipitated in a 100 cc flask with excess of KOH, let stand one hour at 70°, cooled, made up to mark, filtered, and used 50 cc of filtrate for determination. Aluminum was precipitated with ammonium acetate as $AlPO_4$, filtered, washed, ignited, and weighed. Iron was determined by the permanganate method.

Thiosulphate method.

Precipitated according to directions already given. Ignited and weighed the precipitate without treating it with KOH.

Comparison of results.

Methods.	Solution No. 1.		Solution No. 2.		Solution No. 3.	
	Fe_2O_3.	Al_2O_3.	Fe_2O_3.	Al_2O_3.	Fe_2O_3.	Al_2O_3.
	Gram.	*Gram.*	*Gram.*	*Gram.*	*Gram.*	*Gram.*
Theory	0.0207	0.0558	0.0602	0.0239	0.0179	0.0159
Acetate method:						
Single precipitation	.0262	.0560	.0677	.0240	.....	.....
Double precipitation	.0246	.0560	.0597	.0228	.....	.....
Potash method	.0207	.0580	.0594	.0221	.....	.....
	.0200		.0602			
Thiosulphate method	.0207	[1].0535	.0594	[1].0249	.....	[1].0165
	.0210	[1].0563	.0602	[1].0249	.....	[1].0165
	.....	[2].0523	.....	[2].0236	.....	[2].0151
	.....	[2].0535	.....	[2].0228	.....	[2].0150

[1] Precipitated in presence of acetic acid.
[2] No acetic acid. Dissolved in KOH and reprecipitated.

NOTES OF ANALYSTS.

T. S. Gladding, New York.—The Gladding method, as published, was employed with one modification, as follows: An aliquot, 50 cc, of the solution (of 4 grams in 200 cc), containing 1 gram, was placed in a 200 cc flask, 20 cc of 50 per cent solution of c. p. KOH added, digested for thirty minutes in boiling water bath, cooled, filled to mark, mixed, filtered, and 100 cc of filtrate, equal to one-half gram, treated as directed. The complete analysis requires only from one and one-half to two hours when pump is used.

C. Glaser, Baltimore.—Have modified my method as follows: (Chem. Zeit., 1897, 21, No. 69.) The solution of the phosphate is freed from lime as much as possible by means of a precipitation by ammonium acetate, or by precipitation of calcium sulphate, according to the method of E. Glaser. The solution, freed from lime, is approximately neutralized. After cooling, dry sodium peroxid is added, in small portions, until a clear solution is obtained; from 3 to 6 grams will usually suffice on 0.4 gram of substance. Now boil for a short time, let settle, and pour through a thick ashless filter. In the filtrate determine alumina, after acidulation, by any convenient method, preferably by precipitation twice, as $AlPO_4$ (after the addition of a little pure phosphoric acid), by means of ammonium acetate.

The iron upon the filter is dissolved in warm, dilute HCl, and after purification by first precipitating it with nitroso-β-naphthol or ammonium sulph-hydrate, the latter in the presence of citric or tartaric acid in ammoniacal solution, the precipitate is dissolved in acid, oxidized, precipitated as hydroxid, and weighed as oxid.

F. S. Shiver, South Carolina.—Worked acetate method according to Bulletin 21, page 5, South Carolina Experiment Station.

Referee.—The results are too few by any of the methods to admit of much comparison or the drawing of very definite conclusions. There are more results reported on iron by the permanganate method than by any other, and with a few exceptions they agree fairly well. This seems to be the best method for the determination of iron in phosphates. The most promising method for alumina, in my opinion, will be found to be that of Gladding—the potash method.

I would recommend that the work be continued next year.

TABLE V.—*Results of analysis of basic slag.*

Analysts.	"Available" by ammonium citrate.				Available by Wagner's method.		Solubility in 1 per cent citric acid at about 20° C.			
	½ hour.		1 hour.				1 hour.		2 hours.	
	No. 6.	No. 7.	No. 6.	No. 7.	No. 6.	No. 7.	No. 6.	No. 7.	No. 6.	No. 7.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
John Fields, Okla.	.....	.....	.....	.....	[1]11.05	[1]12.81	6.96	7.47	7.70	8.56
					[1]11.05	[1]12.89	6.83	7.52	7.67	8.34
					[2]11.74	[2]14.38	6.88	7.52	7.70	8.68
					[2]11.74	[2]14.48				
C. Glaser, Balto.	.....	[3]6.67	.....	[3]7.37	[1]11.11	[1]13.72				
		[4]6.91		[4]7.75						
H. E. Curtis, Ky.	[5]5.43	[6]6.62	[5]5.98	[6]7.32	.....	.....	7.52	8.14	7.92	8.78
E. G. Runyan, U. S. Dep. Agr.	6.70	7.72	6.53	8.16	[7]11.76	[7]14.34	[8]7.45	[8]7.55	[8]8.03	[8]8.74
Huston and Jones, Ind.	5.34	6.10	5.62	6.89	12.04	24.46	7.59	8.25	8.09	9.49
	5.39	6.03	5.68	6.89	11.97	14.46	7.52	8.44	7.92	9.49
		6.10					7.52			
		6.12								
C. W. Norris, Pa.	.....	.....	.....	.....	[1]11.11	[1]12.95	[9]8.34	[9]9.31	[9]8.73	9.67
					[1]11.15	[1]13.05	[9]8.22	[9]9.10	[9]8.85	9.72
								[9]8.99		
Veitch, Md.	6.72	8.93	7.05	9.54	[11]11.85	[11]13.90	[10]9.81	[10]11.64	[10]9.98	[10]12.72
	6.61	9.16	7.09	9.44	[11]11.85	[11]14.30	[10]9.62	[10]11.56	[10]9.96	[10]12.68
		8.87				[11]14.27				[10]12.49
					[11]11.94	[11]14.04				

[1] Shaken by hand 6 times per minute.
[2] Shaken by hand continuously.
[3] Molybdate method.
[4] Citrate method without reprecipitation.
[5] By difference, association average for total.
[6] By difference, Skinner's total.
[7] At 29° C.
[8] At 28° C.
[9] Citrate method.
[10] 20° C.
[11] Refined two hours for complete precipitation.

TABLE V.—*Results of analysis of basic slag*—Continued.

Analysts.	Mechanical analysis.									
	No. 6.					No. 7.				
	Retained by ½ mm. sieve.	Pass above. Retained by silk, 4 meshes to mm., No. 5.	Retained by silk.	Pass silk, 4 meshes to mm., No. 13.	Pass silk No. 20.	Retained by ½ mm. sieve.	Retained by silk, 4 meshes to mm., No. 5.	Retained by silk, 5 meshes to mm., No. 13.	Pass silk No. 13.	Pass silk No. 20.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
E. G. Runyan, U. S. Dep. Agr.	0.03	0.10	43.14	56.53	.....	4.25	3.66	41.79	50.14	
Huston and Jones, Ind	.07	.17	[1] 29.84	[1] 68.66	.....	[1] 2.86	[1] 1.73	[2] 32.67	62.74	
			[1] 31.11	[1] 69.92		[1] 3.25	[1] 1.66	[2] 32.05	63.04	
			[2] 25.16	[2] 74.60						
			[2] 25.44	[1] 74.56						
Laughlin, Md	.00	.00	28.80	71.20	54.72	3.66	4.13	39.70	52.51	41.08
										41.04

Analysts.	Removed by magnet.		Total P_2O_5.	
	No. 6.	No. 7.	No. 6.	No. 7.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
E. G. Runyan, U. S. Dep. Agr	1.96	0.83	16.08	18.07
Huston and Jones, Ind	[3] 13.13	[4] 4.33	16.20	18.21
	[4] 10.19	[4] 4.96	16 21	18.10
	[4] 10.06	[4] 4.39		
	[4] 11.01	[4] 5.46		
Veitch (chem.) and Laughlin (mechanical anal.), Md	.....	[5] 81.25	.....	[6] 18.12
		[5] 81.34		
	[7] 2.17	[7] 1.13		

[1] Shaking sieve.
[2] Rubbed through with camel's hair brush.
[3] Dipped into slag.
[4] 100 times across at 1½ mm.
[5] In contact.
[6] Skinner.
[7] Not in contact

The slags used in these experiments were analyzed for me by Mr. H. C. Sherman, then of my laboratory staff, with the following results:

Analyses of slag.

Constituents.	No. 6.	No. 7.
	Per cent.	*Per cent.*
SiO_2	5.95	8.19
P_2O_5	[1] 16.30	[2] 18.12
S	.25	.23
CO_2	1.82	1.29
FeO	13.42	11.19
Fe_2O_3	[3] 6.20	5.61
Al_2O_3	[1] .60	1.86
MnO	4.84	4.40
CaO	46.22	43.58
MgO	1.22	.81
H_2O	.62	.35
Total	97.44	95.73

[1] Veitch. [2] Skinner. [3] Based on Veitch's total iron.

NOTES OF ANALYSTS.

H. A. Huston.—From the results obtained and the observations made during the work I am of the opinion that the work with the magnet is of very little account. If the magnet be allowed to come directly in contact with the slag, practically all of the slag will adhere to the magnet. * * * Odor of H_2S was given off in all cases upon uncorking flasks after treating slag with citric acid or acid citrate.

Referee.—It is also the opinion of the referee that the work with the magnet is of no account. Also that the mechanical analysis is not of much value, unless, perhaps, a system of determining available might be based on such determinations, taken in connection with the determination of total phosphoric acid.

The results with the official citrate of ammonia are somewhat discordant; also those with citric acid. The results by the Wagner method are very good considering the various methods used for agitating the flasks. If it appear that basic slag gives as good results in the field as are obtained by acid phosphates I think the Wagner method is probably the best we have at present.

The referee has no recommendation to make, however.

The president called for papers relating to the analysis of matters containing phosphoric acid.

ESTIMATION OF IRON AND ALUMINA IN MINERAL PHOSPHATES.

By G. W. Lehmann.

It is understood that all reagents used must be strictly c. p., and ashless filters used. Two grams of substance are dissolved in sufficient HCl and one or two cc of HNO_3. Evaporate to sharp dryness on water bath. Take up with a few drops of HCl, filter, and make up to 250 cc. Remove 50 cc (equal to 0.4 gram substance) to 8-ounce beaker. If only a few drops of HCl have been used neutralizing is not necessary, as the few drops are just enough to hold the Ca in solution; should more HCl have been needed, nearly neutralize the 50 cc with NH_4O. Add, carefully, solution of ammonium acetate until precipitate forms, then add 5 cc in excess, stirring vigorously, and keep for twenty minutes at a temperature *not exceeding* 70° C. Filter, wash well with tepid water; dissolve residue on filter with warm dilute HCl into 8-ounce beaker, nearly neutralize with NH_4O; increase volume in beaker, if necessary, by adding water to about 75 cc, and add carefully in four or five portions about 2 grams sodium peroxid. This quantity of sodium peroxid will, in most cases, be sufficient to render the solution for a moment perfectly clear and of red-brown color. Place beaker on asbestos plate and boil at once for two or three minutes, when all the Fe_2O_3 will be precipitated and collected at the bottom of the beaker. Filter and wash with hot water. To filtrate, add a few drops of microcosmic salt solution, add HCl until acid nearly neutralizes with NH_4OH, add ammonium acetate, proceed as stated before, repeat this process a second time with the exception of this time adding a few drops of P_2O_5 solution in place of the microcosmic salt; filter, incinerate as usual ($AlPO_4$).

The red Fe_2O_3 precipitate on filter is dissolved with warm dilute HCl. Add to filtrate sufficient concentrated citric acid solution to hold Fe_2O_3 in solution (about 5 cc); make strongly ammoniacal; add about 10 cc strong ammonium sulphid solution, stir vigorously for two minutes, allow to settle, filter, and wash very thoroughly with ammonia water to which a little $(NH_4)_2S$ solution has been added until every trace of citric acid is removed. Dissolve the iron sulphid on filter with warm dilute HCl, add a few drops of HNO_3, boil, add NH_4OH, boil to facilitate quick filtration, let settle, filter, incinerate as usual (Fe_2O_3). Time, about three hours.

Mr. HUSTON. I would especially request the referee that, in connection with the complete analyses of these slags which he quoted, he give us the minutest details of the method of analysis. I have tried every published method which I can find for the complete analysis of slag, and in the hands of myself and my assistants they are all absolutely worthless. They give manganese strung along over the whole course of analysis, and we have not been able to remove that difficulty. I notice that no titanium is reported. One slag, at least, contains titanium, which interferes very seriously with the determination of iron by any volumetric method. The electrolytic methods for the determination of iron are quite worthless with these materials. In fact, we only disintegrated our platinum electrodes without being able to do anything with the method. Yet it is only by the electrolytic method that we have been able to get anything approaching a satisfactory determination in these slags. With that we have, we think, got something which we can get twice alike, at any rate. We have not yet been able to overcome the trouble with manganese. I would not be surprised if we found this same difficulty with other phosphates in which iron is so high that it can not all be precipitated as the ordinary phosphate that we find in the acetate method. Here we have much more iron than we are accustomed to meet with, and it requires enormous quantities to add to this solution to precipitate all the phosphorus, if it be desired to get rid of it that way. Even acid precipitation with calcium sulphate carries a large quantity of manganese with it in this work. So I especially request that we be given the minutest details of the analysis of this material. It is of the utmost importance in connection with the analysis of slag that we should know of what this slag is composed. I believe the percentage of manganese in these slags is going to play an important part in connection with the available phosphorus in them. We will finally, I think, have to know with accuracy the composition of these slags, and, so far as I have been able to find methods, none of them is satisfactory.

In regard to the matter of the neutralization of the citrate solution, we have tried the method spoken of, which is, as I recall it, letting things stand around for twenty-four hours and it is then neutral. The first trial we made of this was highly satisfactory, although I confess we used our calcium chlorid method to determine whether it was neutral, but a number of other trials was decidedly unsatisfactory. The things were very far from neutral, even with litmus paper, but men who were doing research work on citrate solutions in my laboratory were entirely unwilling to work with such solutions.

Mr. SCOVELL. We do not claim that we have any exact method for determining available phosphoric acid in basic slag, but we find we get more concordant results and a smaller percentage if we treat our yellow precipitate at 40° instead of 65°. The old method of heating the

precipitate was at 40° and we changed it to 65°, but if it be heated at 40° it will do better. In fact, I am making some experiments, on which I am not ready to report, in which we precipitate in the cold and we find still better results. It will settle in five or ten minutes. I find this the quickest and easiest way to do it.

Mr. HUSTON. I would like to discuss a little more this matter of work with a magnet on these slags. I suppose the idea of treating slag with a magnet is to remove the actual iron there, and I presume the idea was imported from Europe along with the slag. The determination of what is actually removed from the slag by the magnet shows that that material contains from 8 to 12 per cent of phosphoric acid—the material which actually adheres to the magnet. In our method, the slag is put on a glass plate, and the magnet, which is about the size of a lead pencil, is placed so that it can not come within 1.5 millimeters of the slag. It is then passed over it fifty times in one direction and fifty times crosswise. The operation is tedious, but it works after a fashion. Yet we found that even with these precautions from half to two-thirds of the phosphorus was removed by this method, so I think the use of the magnet is not at all desirable. I think it should be abandoned altogether. The actual amount of real metallic iron I think is very small.

In regard to the fineness of the sample, especially where material like bolting cloth is to be used, I think very explicit directions should be given as to how it is to be used; whether we are to shake the material, or whether we are to brush it. It makes a great difference in determining the fineness, and I believe we should adopt some very explicit directions.

The matter of the solution of slags in citric acid I think is a rather difficult one. As we have already shown, in about one and a half to two hours the amount of phosphorus dissolved reaches a maximum and afterwards falls off rapidly, while the acid continues to be neutralized. If we are working at higher temperatures the maximum amount of phosphorus dissolved is reached at a much earlier point. In view of the very complicated composition of citrates of iron, it seems to me there is very little hope of arriving at any concordant results or anything suitable for a commercial method by treating slags with citric acid, although it has been very strongly recommended. This brings us right back to the position that we shall finally be forced to value slags upon the fineness and total contents of phosphoric acid. In connection with the method just discussed for determining iron and aluminum, we would have again to look for the titanium. I believe that the volume of the solution has a great deal to do with the amount of phosphorus removed. We have done a good deal of work on this subject. It is not final, and I am not yet ready to report on it. Dr. Wiley has his little joke on me for dissolving slag in water; but it is not so funny as it seems to be, for a considerable quantity of basic slag will dissolve

in water. I believe the difference between the official citrate we use and the Wagner method is very largely due to the relation between the amounts of slag taken and the volume of fluid used. It is due more to that than to the acidity of the solution. We can very much increase the amount of phosphorus removed from basic slag by the use of the official solution if we simply add the citrate solution's own volume of water to it. We are now working upon that, trying to get something a little more definite than we have already arrived at. I believe it is much more a question of volume than we have heretofore supposed.

Mr. WILEY. One remark made by the referee I would like to call attention to, and that is that the effect of the mechanical condition of the slags is of little importance. Now, it seems to me that with all these materials, not only basic slags but other mineral substances used as fertilizers, the mechanical condition is of the greatest importance, as the amount of surface which is exposed by the material is of course proportionate to the size of the particles. The finer the mechanical condition, the greater the surface exposed. Now, the solubility of a body is to a certain extent a function of its surface. Very important modifications of solubility with a given solvent are determined by the surface of exposure. So it seems to me that that remark of the referee will bear criticism. The mechanical condition of a slag is one of the first things to be determined. It is true that slag particles when placed in the soil tend to rapid decomposition, due chiefly to the slacking of the lime they contain. It is evident, however, that the finer the slag is at first the quicker will this decomposition take place. In easily soluble substances the mechanical condition is of less importance. I doubt not that the function of water as a solvent is determined largely by the surface of the bodies which are exposed. So far as that is concerned, we know that even gold and platinum will dissolve in water, at least if they contain a little salt. Gold has been found in solution in the waters of the ocean.

I do not know how we are to be sure that phosphatic slag dissolves in water alone. If I understand the composition of basic slag, it has a great deal of lime in it, and when water is added hydrate of lime is produced. The simple addition of water to powdered slag does not prove that water is a solvent, because what we have there is hydrate of lime in partial solution.

This matter of slag phosphate is one of the most interesting now before this association. We ought to introduce those new things which promise so much benefit to agriculture. I think we have reached a degree of accuracy in the analysis of ordinary nitrogen, phosphoric acid, and potash-containing bodies, which is sufficient for all practical purposes, but here comes a substance which presents some unusual peculiarities. We have the remarkable fact presented that when lime is in this certain state of combination it seems to be peculiarly assimilable by plants, and here we are brought directly in contact with the great

point of union between pure chemistry and physiological chemistry in the life of the plant.

I am sorry Congress saw fit to lay a duty of $1 a ton on imported phosphatic slags. All along our farming interests have been favored, and I think justly so, in our legislation. The attempt has been made to bring in, free of duty, all those substances which are used for fertilizing purposes. I fear this duty will delay the large introduction of these phosphates. We have only one or two mills in this country, I think, which are making slag phosphate. They are making steel by the basic process, and of course are producing large quantities of slag, which will, in the course of time, be on the market. Our members will sooner or later be brought into contact with these fertilizing materials, and it may be that there will be presented to us problems different from those which we have been engaged upon. We should not only welcome the results that have been obtained in these matters, but the referee for next year should be instructed to continue the investigations with regard to the solubility of basic slags and to their properties as plant food. The experience of ten years is not to go for nothing. It has been certainly proved that these slags have a high agricultural value. I would like to see the association push forward this work. We are not in condition to adopt any method as official. As the results show, the Wagner method has given the most concordant results among a number of analysts, and we can not do better than hold on to it as a provisional, not an official, method. Later we may reach a position when we can adopt an official method which will be enjoined upon us all, but this, I think, is not possible at the present time. This whole matter seems to me to bring up a subject which we must sooner or later investigate to the bottom—namely, the true availability of phosphatic manure. Many statements and claims have been made in regard to this matter. Soft ground phosphate has been put upon the markets of this country with the claim that it is the only natural plant food. On the other hand, it has been claimed that the phosphoric acid in ground bone is worth nothing as plant food. Here we have two extremes of opinion. I know by actual experience that soft ground phosphate in some soils is efficient in feeding plants, while in others it is practically worthless, and I have a very firm conviction that the phosphoric acid of ground bone is more or less available for the uses of plants.

Mr. McDonnell. What Mr. Wiley says in regard to slag is very interesting, but I think what I said was not understood. I did not mean that it made no difference whether the slag was fine or coarse, but I meant that if we determined the availability of the slag by treating with some of these reagents, the finer the slag the more would be dissolved and the availability would be shown by the amount dissolved, without the necessity of making a mechanical analysis. If the slag be very coarse, the soluble action will be very much less, depending upon the amount of surface exposure. So I hardly thought it worth while to have both methods of determination used.

The point Mr. Huston mentioned about the use of the magnet we had observed in our laboratory before we received his report, and it will be noticed that in two determinations, Table No. 5, made in our laboratory, in the first one we removed by a magnet over 81 per cent of the slag (No. 7) where the magnet was brought in actual contact with the substance, while when it was held a very short distance away from the substance only a little over 1 per cent was removed.

Mr. GAINES. Mr. Wiley said in the course of his remarks that ground soft phosphate had been found to act well in certain soils. At the Virginia department of agriculture we receive many samples of that ground soft phosphate, the farmers stating that it is absolutely worthless. I would like Mr. Wiley to state the peculiarities of the soils in which soft phosphate was found to respond.

Mr. WILEY. I do not know whether the material could be called soil or not. It was the peat or muck soils of Florida, extremely acid and composed almost exclusively of organic matter. In experimenting with these soils in pots I found I could detect very little difference between superphosphate and soft ground phosphate. They all seemed to act very happily.

Mr. PERSONS. In Florida we have conducted experiments that corroborate Mr. Wiley's, and the best effects were always produced in soils containing a large amount of organic matter. In mixing fertilizers it was found that where soft phosphate was used in the presence of cotton-seed meal the results would seem to be borne out there also. That is, where nitrogen was used in organic combination, the effects of the soft phosphate were much more perceptible. In my mind that is hardly a question any more; it is almost entirely settled. If we get good effects from soft phosphate it is when it is applied along with one of these soils containing large amounts of organic matter.

The PRESIDENT. The fact referred to was in the mind of the Chair this morning, that we might eventually be obliged to classify methods of determinations for agricultural purposes, with reference to the classes of soils upon which the fertilizers were to be applied.

Mr. MCDONNELL. In regard to the factor for calculating results on phosphoric acid, I wish to mention that in some sections I think the tables of Battle and Dancy are used, which give different results from the tables published by myself. On some samples in this work this would make a difference of 0.15 to 0.16 per cent between the two tables. So far as I know, the association has never adopted an official factor, but left it to each investigator.

Mr. WILEY. I move that the portion of the referee's report referring to the different methods for slags be referred to the committee, and especially with reference to the adoption of a provisional or an official method for the guidance of our members during the coming year.

Seconded, and so ordered.

The meeting was then adjourned until Wednesday morning.

SECOND DAY.

WEDNESDAY—MORNING SESSION.

The association met at 10 o'clock, and the president appointed Messrs. Penny, Van Slyke, and Fulmer a committee on nominations, to report at the afternoon session at 2 o'clock.

RECOMMENDATIONS AND DISCUSSION OF METHODS FOR PHOSPHORIC ACID.

The committee on recommendations presented the following report on methods of analysis for phosphoric acid:

The committee recommends—

(1) That the volumetric method for determination of P_2O_5 be continued another year as a provisional method, and that the referee for next year be asked to continue work on this method.

(2) That a continuation of the study of methods of analysis of Thomas slag be made.

(3) In regard to iron and alumina, that methods for determination of these substances in phosphate be further investigated.

Mr. BARTLETT. Referring to the referee's first recommendation in regard to the volumetric method, the committee, after thoroughly discussing this method and talking with other analysts who have had experience with it, has decided that it can not recommend it this year to be adopted as an official method, but does recommend that it be continued as a provisional method, and that further investigation be made of it. This is the volumetric method, either the Kilgore or McDonnell modification.

Discussion arose as to whether "provisional" or "optional" were meant in this recommendation. Mr. Van Slyke read the note inserted in the "Methods," page 13, on this point.

The PRESIDENT. Would it not clear up all misunderstanding if the Chair were allowed to state the recommendation as follows:

That the committee recommends that the volumetric method remain stated, as heretofore, as a provisional method, and it be recommended for further study.

Mr. WHEELER. A number of members of the association have stated that they have tried this method and abandoned it, being unable to get good results. Other members have obtained at times good results, and at other times poor results. Others claim they have obtained uniformly good results. It seems to me that so long as there are so many who obtain poor results at times or have had to give it up, it would be

unwise for this association to adopt it until it has been further studied and shown to be reliable in the hands of the average analyst.

Mr. SCOVELL. I think none of us wants to adopt a method that would not give uniformly good results on all phosphates. I would hate to go into court and say I had analyzed a fertilizer by a method I was not certain of for high percentages. Both Mr. Peter and Mr. Curtis were much in favor of this method, and they put in a month's solid work. But they failed with it. They could not trust it in high percentages, although it worked very well on low percentages. They tried it very carefully, but it did not always bring good results. I believe the cause is this, that the precipitate is not uniformly of the same composition, and I think we will have to study out some method by which the yellow precipitate will be of uniform composition. Until that is done I do not think we can use the volumetric method and get theoretical results every time. It would be unwise for this association to adopt it as an official method, although it may be adopted as a provisional method, and it is very worthy of further investigation.

Mr. KILGORE. The method is certainly much more rapid than the old gravimetric one, but I fully agree with Mr. Scovell in his remarks as to its adoption. In the case of this and every other method I should very much prefer to see that it is thoroughly all right before we put it in the "methods."

The recommendation was thereupon adopted.

Mr. BARTLETT. The committee recommends that the study of methods of analysis for Thomas slag be continued.

Mr. HUSTON. I would like to inquire whether this recommendation is for phosphates alone, or for complete analysis, which is a very different thing. I do not know what the committee had in mind, but I hope it was complete analysis rather than simply examination for total phosphoric acid.

The PRESIDENT. The Chair would rule that it is not intended in this case to confine the referee or the association to this particular line of work (phosphoric acid).

The recommendation was thereupon adopted.

Mr. WILEY. While I am far from believing in the so called Wagner method as the only and sole method for analysis, it is evident that we can get comparative results by it. Would it not be well for the association to recommend it as a provisional method while this study is going on, in case it is called upon to examine these slags officially, as it may be before another year? If these slags come on the market before another meeting, any or all of our members may be brought into contact with them officially, and in that case a provisional method recommended by this association would be the only thing they could rely upon.

The PRESIDENT. The committee will take that matter into consideration and report upon it.

Mr. HUSTON. I would like to say a word on this subject. I suppose we are after the available phosphoric acid in this slag. First, have we any evidence whatever in any slag upon which any of the members of this association have worked that there is more than one compound of phosphoric acid present? Is there more than one kind of phosphoric acid in it? Is that phosphoric acid combined with 4 molecules of lime, or is there some other form than what I suppose we will have to call the ortho-phosphoric acid at present? It seems to me that all the methods which we have or have considered simply take out a part of the phosphoric acid. They do not distinguish between phosphoric acid combined with 4 molecules of lime and any other form. This is particularly true of our present official method with neutral citrate. If our official citrate solution be merely diluted and 100 cc put in 100 cc of water, and 2 grams treated with it, very much more phosphoric acid will be dissolved from this slag. If half a gram be used, a still larger per cent will be dissolved. In fact, this is true for any phosphate outside of any material like hard rock. I think it is very largely a question of volume, and, to some extent, of the quantity of reagent used. In examining some slags, two of which were the association's samples, to see if it were possible to work out any scheme by which we could tell how much of this tetrabasic phosphate is present, I have run down some slags, and if my theory in the matter be right all the phosphorus in them is present as the tetrabasic compound. If this be so, I do not see why the part which the citrate happened to miss is not just as good as the part which the citrate did dissolve, the trouble there being simply that we did not use enough volume or did not use fluid enough to keep the resultant product in solution.

So I believe we ought to exercise a little care about adopting the method. I realize, of course, that the Wagner method may give higher results than the official method, but, notwithstanding the fact that they are said to approximate agricultural results, I think we ought to be a little slow about adopting a method for the examination of these slags. I think it would be far safer at present, with the limited amount of slag that there will be on our market and the very uncertain character of it as it may come from some of the works, either to adopt the total phosphoric acid as the standard, or at least put it beside the other. Neither, as I understand it, is to be the official method.

Mr. LINDSEY. I want to call attention to the fact that Wagner has made a great many experiments regarding the availability of slag on various kinds of agricultural plants, and he has shown that it has a certain degree of availability. Although I have not given his method critical study, yet my impression is that it corresponds very well with actual results which he got from plant experiments. If we are going to approve a method for determining the availability of phosphoric acid in material of that kind, I believe Wagner's method is better adapted than anything else that has been provided, and it seems to me wise that

we should adopt that method provisionally until something better is offered.

Mr. WHEELER. The important point to consider is that we are liable to have European slag placed upon our market before the close of this year. There is much surplus slag in Europe, and they are anxious to bring it over here. If it be analyzed by our citrate method, which we have to use under present circumstances, it seems to me the farmers of this country will be led to think that it is less valuable for agricultural purposes than it really is. The Wagner method shows a higher percentage of available phosphoric acid and comes nearer to agreeing with actual experiments on plants, and there is no reason why we should throw a wet blanket on this most excellent material. For that reason it would seem to me much better for the association to use this method.

The PRESIDENT. If there be no further remarks, the matter will be considered as referred to the committee on recommendations.

Mr. MCDONNELL. Before we leave this question I think the methods for determining iron and aluminum ought also to be referred to the referee on phosphoric acid for next year. I make a motion to that effect.

Mr. Kilgore moved that it be referred to the committee on recommendations. Adopted.

Mr. Bartlett stated that the committee on recommendations was anxious to finish its work, and if in order, would complete its recommendations on phosphoric acid with regard to basic slag.

Mr. Lindsey's motion that the report be heard was carried.

Mr. BARTLETT. This recommendation refers to the determination of phosphoric acid in basic slag. The committee recommends that a description of the Wagner method be inserted in the "Methods" as a provisional method for determining phosphoric acid in basic slag. Adopted.

Mr. HUSTON. I wish to urge members to work a little more on slags. The method calls for a certain fixed temperature, I believe 15° or 17°. I have learned from some workers who come here that they find this temperature of the Wagner method either impossible or very inconvenient, and that they have departed from it. This fact may have prevented others from working on it, and I wanted to say that in basic slag the question of temperature is practically nothing. If you will recall what I told you last year, you will see that basic slag differs radically from nearly every other known phosphate in that it makes very little difference what temperature you use, from 4° to 80°, so if any of you have been prevented from taking up the work by the requirements of temperature, I will say that the question of temperature has practically no bearing on the value of the work which you may do.

The report of the committee on recommendations was then called for.

Mr. BARTLETT. The third recommendation of the committee is:

In regard to the determination of iron and aluminum the committee recommends that methods for the determination of these substances in basic slag be further investigated.

Mr. SCOVELL. I think it was the intention that the recommendation cover iron and aluminum in phosphates. I move that the report of the committee be so amended.

The amendment was adopted.

The PRESIDENT. The question is now upon the motion as amended, that the study of iron and aluminum in basic slags and phosphates in general be continued.

The recommendation was adopted.

Mr. HUSTON. What action was taken in regard to the Wagner method for slags? My recollection is that it was decided to publish the method, in order that all might have an opportunity to try it if they wished. The most conspicuous reason given for publishing the Wagner method is that it gives higher available than our official method. Now, that is not a good reason. I do not think it is the main reason. The main reason is that it has been extensively used in Europe, and it is said that it approximately follows the results derived from plant or pot experiments in the use of this material. It is a matter of great importance where we publish that method. I think we ought to be very careful not to put it in the official methods of the bulletin, but in the volume of the proceedings. If you get it inside the covers of the "Methods," labeled an official method, you will never get it out, and you will have no end of trouble with people who have slag to sell. It is much easier not to put it in than it is to get it out. I wish to guard the matter carefully. If there be any intention to put it among our official methods, we had better put it where it will be easily accessible, but do not let it be supposed that we have given it our official sanction.

It was moved and seconded that the action taken with reference to the Wagner method be reconsidered. Carried, and the matter referred to the committee on recommendations.

Mr. Persons asked for a definition of "optional" and "provisional," as applied to methods.

The PRESIDENT. The word provisional, as contrasted with optional, has this meaning: That the association is not prepared to recommend officially a method as official, but that in the absence of any method with which it is satisfied it suggests this as one which may be used for the purpose. A member uses it on his own responsibility. It is not regarded as an official method.

Mr. BARTLETT. The committee recommends that a description of the Wagner method for the determination of phosphoric acid in basic slag be inserted in the proceedings of the association, and that a note be inserted in the "Methods of Analysis," under the head of "Phosphoric acid," to the effect that the Wagner method may be found on page 72 of the said proceedings.

Mr. HUSTON. I think it would be well to make it "the Wagner method for determining the citrate soluble," and I move to amend to that effect.

The amendment was seconded.

Mr. LINDSEY. That is, merely publish it in the proceedings as Wagner's citrate soluble method; not as an official or provisional method. Do I therefore understand that we shall proceed with slags to use the official method, namely, ammonium citrate solution, for the determination of the available phosphoric acid in slags? Do I understand that to be the sense of this meeting?

The PRESIDENT. In Bulletin No. 46 the general statement is, "Method for the examination of fertilizers," and under "Determination of phosphoric acid" there is nothing said with reference to the source of that phosphoric acid, indicating that basic slag is not included. In that case it would seem that the official method would remain the same as before.

Mr. Huston thought this would depend largely upon the ruling of the statutes under which chemists operated. In his own State the statute says, "soluble and reverted phosphoric acid." Each must study the statute in his State and determine what sort of ruling he can make upon it.

Mr. LINDSEY. I want to protest against the official method, whether you call it soluble or available or reverted, for determining the soluble and available phosphoric acid in Thomas slag.

Mr. HUSTON. If you look at last year's proceedings you will find that you can use either solution and get identical results if you will modify the treatment somewhat. Our official solution can be made to do just as the Wagner solution. It is not in the solution; it is in the handling of it. I am not in favor of using either one at present on Thomas slag. I have done a great deal of work, which I did not see fit to report, in investigating whether all phosphoric acid in certain slags is in the form of actual tetrabasic phosphates. If it is, I am convinced it should all have the same valuation. Moreover, our climatic and soil conditions are so different that I do not think we should take any steps toward adopting anything which is based upon climatic conditions in Europe.

The recommendation was adopted and the method is given here.

(The amendment seems to have been lost sight of.)

WAGNER METHOD FOR THE DETERMINATION OF PHOSPHORIC ACID IN SLAGS.

(Principles and Practice of Agricultural Analysis, Vol. II, pp. 78–80.)

Solutions employed—Ammonium citrate.—In 1 liter there should be exactly 150 grams of citric acid and 27.93 grams of ammonia, equivalent to 23 grams of nitrogen. The following example illustrates the preparation of 10 liters of the solution: In 2 liters of water and 3½ liters of 8 per cent ammonia, 1,500 grams of citric acid are dissolved, and the cooled solution made up exactly to 8 liters. Dilute 25 cc of this solution to 250 cc and treat 25 cc of this with 3 grams of calcined magnesia and distill into 40 cc of half normal sulphuric acid. Suppose the ammonia nitrogen found correspond to 20 cc of fourth normal soda lye. Then in the 8 liters are contained $\frac{20.0 \times 0.0035 \times 8000}{2.5} = 224$ grams of ammonia nitrogen. Then in order to secure in the 10 liters the proper quantity of ammonia there must be added 2 liters

of water containing 230—224=6 grams of nitrogen, or 7.3 grams ammonia; viz, 94 cc of 0.967 specific gravity.

Molybdate solution.—Dissolve 125 grams of molybdic acid in dilute 2.5 per cent ammonia, avoiding a large excess of the solvent. Add 400 grams of ammonium nitrate, dilute with water to 1 liter, and pour the solution into 1 liter of nitric acid of 1.19 specific gravity. Allow the preparation to stand for twenty-four hours at 35° and filter.

Magnesia mixture.—Dissolve 110 grams of pure crystallized magnesium chlorid and 140 grams of ammonium chlorid in 700 cc of 8 per cent ammonia and 1,300 cc of water. Allow to stand several days and filter.

The directions for determining the phosphoric acid in slags and raw phosphates soluble in citrate solutions are as follows:

Five grams of the material, as it is sent into commerce without grinding or sifting, are placed in a half-liter flask and covered with nearly a quarter liter of water, and then 200 cc of citrate solution added, prepared as described above. The flask is filled to the mark with water. The flasks are closed with rubber stoppers and without delay placed for half an hour in Wagner's shaking apparatus. On removal from the apparatus the mixture is filtered and 50 cc thereof treated with double the quantity of molybdic solution at 80°, and the precipitate separated after cooling. The precipitate is carefully washed with 1 per cent nitric-acid mixture, after which the filter is broken and the precipitate washed into a beaker with 2 per cent ammonia and the filter washed therewith until about 100 cc have been used. If the solution be turbid from the presence of silicic acid, it should be precipitated a second time by addition of molybdic solution until the acid reaction is restored. The ammoniacal solution of the yellow precipitate is treated, drop by drop, with constant stirring, with 15 cc of magnesia mixture, and set aside for two hours. The precipitate is collected, washed, ignited, and weighed in the usual manner. The direct precipitation of the phosphoric acid by the magnesia solution in presence of citrate is not advisable, because of the almost general presence of silicic acid, which would cause the results to be too high.

The chair at this point introduced C. E. Munroe, dean of the Graduate School of Columbian University, who welcomed the members of the association to the university and made a cordial tender of its facilities.

Mr. Ewell exhibited and explained a piece of apparatus he had devised for the determination of nitrogen by a modification of the Schultze-Tiemann method.

The chair called for the report of the referee on soils and ash (Professor Goss). The report was read by Mr. Wiley, as follows:

REPORT ON SOILS AND ASH.

By ARTHUR GOSS, *Referee*, and HARRY SNYDER, *Associate Referee*.

As the first step in the soil work this year, the following circular letter was sent out to the chemists of all the experiment stations of the United States and to such others as it was thought might be interested in the work:

MESILLA PARK, N. MEX., *February 16, 1896.*

DEAR SIR: The referees on soils and ash for the A. O. A. C. would be very much pleased to have you take part in the work this year.

In accordance with the instructions given the referee by the association at the last meeting, the following points will be taken up in the soil work:

(1) A further study of the N/5 HCl method, outlined in last year's proceedings of the A. O. A. C., for the determination of available phosphoric acid.

(2) A study of the calcium and ammonium chlorid methods, suggested last year, for the determination of available potash.

The above methods gave some very promising results last year, and it is thought that the work this year may be of considerable interest.

(3) A study of the nitrogen contents of the humus as a means of determining the availability of nitrogen.

(4) In additon to the above it has been thought desirable to call for a determination of total potash, phosphoric acid, and nitrogen in the samples, for the purpose of comparison with the presumably available found.

Through the kindness of Mr. H. W. Wiley and Sir Joseph Henry Gilbert, arrangements have been made by which samples of some of the Rothamsted soils will be secured for this work. The samples will be distributed from Washington.

The ash sample will be of a native New Mexico plant of common occurrence. It is hoped that the ash work this year may lead to some much needed changes in the methods of analysis.

Working directions will be sent to those who take part in the work.

Kindly notify the referee if you desire the samples.

Very respectfully,

ARTHUR GOSS, *Referee.*
HARRY SNYDER, *Associate Referee.*

"Regrets" were received from a considerable number of chemists, and requests for samples from the following: H. A. Huston, Lafayette, Ind.; Harry Snyder, St. Anthony Park, Minn.; G. L. Teller, Fayetteville, Ark.; F. W. Traphagen, Bozeman, Mont.; W. R. Perkins, Agricultural College, Miss.; F. W. Woll, Madison, Wis.; H. B. Battle, Raleigh, N. C.; C. G. Hopkins, Urbana, Ill.; A. D. Selby, Wooster, Ohio; J. A. Widtsoe, Logan, Utah; H. B. Hardin, Clemson College, S. C.; E. F. Ladd, Fargo, N. D.; G. C. Caldwell, Ithaca, N. Y.; Elton Fulmer, Pullman, Wash.; A. L. Emery, San Francisco, Cal.; Arthur Goss, Mesilla Park, N. Mex.; F. T. Shutt, Ottawa, Canada.

Upon the arrival of the soil samples from England samples were sent to each of the chemists requesting the same, accompanied by the following working directions:

DIRECTIONS FOR WORK ON THE A. O. A. C. SOIL AND ASH SAMPLES FOR 1897.

THE SOIL SAMPLES.

Should you not find time to complete all the work outlined below, kindly report such results as you may secure.

Test all reagents used by running blank determinations with at least the quantities used in the regular determinations.

Calculate all results to the water-free basis.

(*1*) *Litmus-paper test.*—Test the reaction of the soil with litmus paper. See Eighth Annual Report of the Rhode Island Experiment Station, pages 232 to 280.

(*2*) *Determination of moisture.*—Use the official method, page 38, Bulletin No. 46, Division of Chemistry, United States Department of Agriculture.

(*3*) *Determination of phosphoric acid by treatment with N/5 HCl.*—Prepare a N/5 solution of HCl by titrating against a standard KOH solution containing little or no carbonate. Use phenolphthalein as the indicator.

(*a*) Digest 20 grams of soil with 200 cc of the N/5 acid in a water bath kept at 40° C. for exactly five hours. Filter off the solution through a dry paper until clear, and titrate 20 cc of the filtrate with standard carbonate-free KOH solution, using phenolphthalein as the indicator. From the data thus secured calculate the amount of HCl necessary to be added so that the solution may be N/5, after allowing for the acid neutralized.

(*b*) Transfer 100 grams of the soil to a half-gallon glass-stoppered bottle or other similar vessel, and add exactly 1,000 cc of N/5 HCl, corrected for neutralization as in (*a*). (See last year's Proceedings.) Place bottle in a large water bath (a metal washtub will answer) and keep at a temperature of 40° C. for exactly five hours, shaking thoroughly each half hour. At the end of the digestion shake contents of bottle well and pour quickly upon a large, dry, ribbed filter of two thicknesses of paper and of sufficient size to receive the entire contents of the bottle. The filtrate is to be received in a dry vessel and the solution poured back through the paper until entirely free from soil particles. Evaporate 500 cc of the filtrate to dryness in a porcelain dish after adding a few cc of nitric acid to oxidize organic matter, moisten the residue with HCl, take up with water, and filter into a flask of about 450 cc capacity. Add 15 grams of ammonium nitrate in solution, add strong ammonia until a permanent precipitate forms, and then concentrated HNO_3, until the precipitate

dissolves. Dilute the solution to about 100 cc if not already to that volume, heat to 85° C., add 75 cc of recently prepared molybdate solution, digest in a water bath at 80° for fifteen minutes, with occasional shakings, and continue the determination as given in Bulletin No. 43 of the Division of Chemistry, United States Department of Agriculture, pages 58 to 60.

(4) *Determination of potash by treatment with alkaline ammonium chlorid (Huston).*—Make up an N/20 solution of ammonium hydrate by titrating against standard acid, using cochineal as the indicator, and dissolve 10.68 grams of c. p. crystalline NH_4Cl in each liter of the same.

Digest 100 grams of soil with 1,000 cc of this solvent exactly as under (1). Evaporate 500 cc of the filtrate to a small volume in a porcelain dish, transfer to a good-sized platinum dish, add one-half cc of concentrated H_2SO_4, and finish the evaporation over the water bath. Heat in the air bath at 140° to 150° C. until danger from popping is past, ignite off the ammonium salts and organic matter, and continue the determination by the Lindo-Gladding method.

(5) *Determination of potash by treatment with N/5 $CaCl_2$ solution.*—Prepare an N/5 solution of $CaCl_2$ by dissolving the c. p. salt in distilled water and titrating against standard $AgNO_3$ solution, using potassium chromate as the indicator.

Digest 100 grams of soil with 1,000 cc of this solvent for five hours at 40°, exactly as under (1). Evaporate 500 cc of the filtrate to dryness in a porcelain dish after adding a few cc of HCl and HNO_3. Take up with water, add ammonium hydrate to alkaline reaction, and precipitate lime, etc., with ammonium carbonate and oxalate. Filter, add one-half cc of concentrated H_2SO_4 to the filtrate, and evaporate to dryness in a large platinum dish. Thoroughly dry in the air bath at 140° to 150° C. to prevent popping, ignite off the ammonium salts, and continue the determination by the Lindo-Gladding method.

(6) *Determination of humus.*—Weigh out either 5 or 10 grams of soil into a 200 cc wide-mouth glass-stoppered bottle. Add 150 cc of a 1 per cent solution of HCl. If the soil contain more than 1 per cent of CaO, then a 2 per cent solution of HCl may be used. Place the stopper in the bottle and shake vigorously. After allowing the acid to act on the soil for five or ten hours the solution is decanted into a filter, a fresh quantity of 100 cc of acid is added, and after about two hours action it is also decanted and the contents of the bottle transferred to the funnel. The soil in the funnel is washed with the dilute acid until no reaction for CaO is obtained, and then with distilled water until the washings are only slightly turbid with $AgNO_3$. After receiving the above treatment the soil is transferred from the funnel to the stoppered bottle, using 150 cc of a 3 per cent solution of ammonia. The bottle containing the soil and ammonia solution is occasionally shaken, and after eight or ten hours the solution is decanted into a 500 cc measuring flask. A fresh quantity of 100 cc of ammonia is added to the soil in the wide-mouth bottle, and after acting on the soil for three or four hours it is decanted and added to the first extract. After the third extraction the extract usually becomes nearly colorless. After the fourth extraction the extract of the humus solution in the flask is made up to 500 cc, and shaken well. After allowing about an hour for the contents of the flask to settle, a 50 cc portion of the filtered humus extract is evaporated to dryness in a tared platinum dish. After drying in either an air or water bath, preferably an air bath at 101° or 102° (on account of the tenacity with which the humus retains water), the dish with the humus materials is weighed, then ignited and weighed again, the loss of weight being calculated as humus.

(7) *Nitrogen content of the humus.*—First treat 5 or 10 grams of soil with dilute HCl in the same way as for the humus determination, then extract the soil with a 3 per cent solution of NaOH in the same way as the ammonia extraction. Two hundred cc of the filtered solution are evaporated nearly to dryness in a Kjeldahl distillation flask, after first making the solution acid with H_2SO_4. The remainder of the acid is added, and the nitrogen determination is completed in the usual way.

(8) *Determination of total nitrogen.*—Use the method outlined on page 2 of Circular No. 2, Chemical Division, United States Department of Agriculture.

Nitrates are not to be included.

(9) *Determination of total phosphoric acid.*—Use the method outlined on page 6 of Circular No. 2, Chemical Division, United States Department of Agriculture. Also see bulletins of the Division of Chemistry, United States Department of Agriculture, No. 43, pages 58 to 60, and No. 47, pages 50 to 57.

(10) *Determination of total potash.*—Use the J. Lawrence Smith method given in Crook's Select Methods, second edition, pages 28 to 40.

The ash sample.—The ash is of Tornillo wood (*Prosopsis pubescens*, Benth).

Analyze by the association method. See Bulletin No. 46, Chemical Division, United States Department of Agriculture, pages 43 to 45.

Please report suggestions as to changes in the methods of analysis.

Respectfully,

ARTHUR GOSS, *Referee.*
HARRY SNYDER, *Associate Referee.*

Up to date the following results on the soil samples have been received:

TABLE NO. 1.—*Soil samples. Results calculated water-free.*

Analysts.	Reaction with litmus paper.	Per cent of moisture.	Per cent of acid neutralized by preliminary digestion.	Per cent total N, official method.	Per cent total N, HgO omitted.	Per cent humus.	Per cent N in humus, with 3 per cent NaOH.	Per cent N in humus, with 3 per cent KOH.
Sample No. 1.—Gilbert mark W3. Wheat continuously for 53 years without any manure.								
C. C. Moore, U. S. Dept. Agr	.....	.....	41.5	.....	.....	2.30	.....	.....
T. C. Trescot, U. S. Dept. Agr	.....	.....	.....	0.09	.....	.....	1.00	.....
A. M. Peter, Ky	.....	1.66	43.7	.....	.....	.....	.....	.....
L. M. Bloomfield, Ohio	.....	1.56	.....	.11	.....	3.29	2.64	.....
John Thompson, S. C	Neutral.	1.41	.....	.12	.....	1.47	4.92	.....
Elton Fulmer, Wash	.....	1.65	.....	.....	.....	.....	.....	.....
J. F. Moore, Ark	.....	1.75	.....	.....	.....	.....	.....	.....
Harry Snyder, Minn	.....	.....	.....	.094	0.088	1.58	3.17	3.17
R. F. Hare, N. M	Alkaline.	1.59	45.2	.10	.....	.....	.....	.....
Arthur Goss, N. M	Alkaline.	.....	44.2	.....	.....	.....	.....	.....
Average	Alkaline.	1.60	43.7	.102	.088	2.16	2.93	3.17

Analysts.	Reaction with litmus paper.	Per cent moisture.	Per cent acid neutralized by preliminary digestion.	Per cent total N, official method.	Per cent total N, HgO omitted.	Per cent humus.	Per cent N in humus, with 3 per cent NaOH.	Per cent N in humus, with 3 per cent KOH.
Sample No. 2.—Gilbert mark B2A. Fertilized for 53 years with 200 pounds ammonium salts and 350 pounds superphosphate per annum.								
C. C. Moore, U. S. Dept. Agr	.....	.....	38.5	.....	.....	2.19	.....	.....
T. C. Trescot, U. S. Dept. Agr	.....	.....	.....	0.10	.....	.....	1.10	.....
A. M. Peter, Ky	.....	1.78	34.5	.....	.....	.....	.....	.....
L. M. Bloomfield, Ohio	.....	1.68	.....	.12	.....	2.93	2.41	.....
John Thompson, S. C	Neutral.	1.65	.....	.12	.....	2.38	3.14	.....
Elton Fulmer, Wash	.....	1.88	.....	.....	.....	.....	.....	.....
J. F. Moore, Ark	.....	2.03	.....	.....	.....	.....	.....	.....
Harry Snyder, Minn	.....	.....	.....	.104	0.095	1.82	3.00	3.00
R. F. Hare, N. M	Alkaline.	1.87	35.7	.11	.....	.....	.....	.....
Arthur Goss, N. M	Alkaline.	.....	35.1	.....	.....	.....	.....	.....
Average	Alkaline.	1.81	35.9	.110	.095	2.33	2.41	3.00

TABLE NO. 2.—*Soil samples—Results calculated water-free.*

Analysts.	Per cent total P_2O_5. Goss method.	Per cent total P_2O_5. Five hours' digestion with HNO_3.	Per cent P_2O_5 with N/5 HCl.	Per cent P_2O_5 with N/5 HNO_3.	Per cent total K_2O. Smith method.	Per cent total K_2O with HFl.	Per cent K_2O with N/5 NH_4Cl.	Per cent K_2O with N/5 $CaCl_2$.	Per cent K_2O with N/5 HCl.	Per cent K_2O with N/5 HNO_3.
Sample No. 1.—Gilbert mark W 3. Wheat continuously for 53 years without any manure.										
C. C. Moore, U. S. Dept. Agr.	0.13	.....	[2]0.008	.....	1.21	.....	0.012	0.014	.....	.....
A. M. Peter, Ky.	.....	.....	[3].004	[3]0.003	.....	.....	.008	.006	0.019	0.019
L. M. Bloomfield, Ohio	.10	.....	.003	.....	.97	.....	.017	.020	.....	.....
John Thompson, S. C	[1].03	.....	.006	.....	1.42	1.38	.010	.007	.....	.....
Elton Fulmer, Wash.	.....	.....	.002	.....	.....	.....	.....	[1].029	.....	.....
J. F. Moore, Ark.	.12	.....	.003	.....	.....	.....	.....	.008	.....	.....
Harry Snyder, Minn.	.12	[2]0.12	.004	.....	.....	.....	.....	.....	.....	.....
R. F. Hare, N. M.	.13	.....	.....	.....	.....	.....	.....	.....	.....	.....
Arthur Goss, N. M.	.11	.....	.005	.....	.....	.....	.....	.....	.....	.....
Average	.118	.12	.0039	.003	1.20	1.38	.0118	.0110	.019	.019
Sample No. 2.—Gilbert mark B2A. Fertilized for 53 years with 200 pounds ammonium salts and 350 pounds superphosphate per annum.										
C. C. Moore, U. S. Dept. Agr.	0.20	.....	0.033	.....	1.08	.....	0.009	0.012	.....	.....
A. M. Peter, Ky.	.....	.....	[3].036	[3]0.029	.....	.....	.005	.003	0.016	0.017
L. M. Bloomfield, Ohio	.21	.....	[1].027	.....	1.45	.....	.020	.013	.....	.....
John Thompson, S. C	[1].09	.....	.037	.....	1.35	1.30	.009	.005	.....	.....
Elton Fulmer, Wash.	.....	.....	[1].054	.....	.....	.....	.051	.020	.....	.....
J. F. Moore, Ark.	.18	.....	.038	.....	.....	.....	.....	.005	.....	.....
Harry Snyder, Minn.	.16	[2]0.16	.033	.....	.....	.....	.....	.....	.....	.....
R. F. Hare, N. M.	.18	.....	.....	.....	.....	.....	.....	.....	.....	.....
Arthur Goss, N. M.	.17	.....	.037	.....	.....	.....	.....	.....	.....	.....
Average	.183	.16	.0357	.029	1.29	1.30	.0188	.0097	.016	.017

[1] Not included in the average. [2] Molybdate precipitate made at 65°.
[3] Molybdate precipitate made at 40° in four hours.

For the phosphoric acid and nitrogen work, the soil samples sent out were probably the best that could have been secured. The great length of time that the Rothamsted experiments have been in progress has made the field data with which to compare the analytical results thoroughly reliable.

For the potash work, it would have been better to have sent out another sample, which had been fertilized with potash, to be used in connection with No. 1, which has received no fertilizer. Neither of the samples sent out has received any potash during the last fifty-three years of continuous cropping. This point was discovered by the referee too late to be rectified; so about all that has been secured this year in the potash work is a comparison of results. In order that the results of this

year may be made more valuable, it would probably be well to send out one of the Rothamsted samples next year that has been fertilized with potash salts.

It was thought by the referees that more could be accomplished by dividing the work. Mr. Snyder very kindly consented to take charge of the nitrogen work, and to him credit for the same should be given. He makes the following comments upon that part of the work:

There is only a very slight difference in the total nitrogen content of the two soils. A marked difference could not be expected because there has been no residual nitrogen to be added to the total nitrogen originally present in the soil. The ammonium salts not utilized by the crops, according to the Rothamsted reports, have never accumulated in the soil for the succeeding crops. Our results are in accord with this fact. A marked difference in the amount of humus in these two soils could not be expected, because humus-forming manures have not been used. The nitrification and oxidation of the humus in these two soils during the successive years of cultivation have undoubtedly been quite uniform. Soil No. 2, although it has received an annual dressing of ammonium salts, is, according to the results, deficient in nitrogen. This is in accord with the field results, because remunerative crops can not be obtained without the continued use of ammonium salts on the plot. On this point the field results and analytical results agree. The per cent of nitrogen in the humus from both plots is low—much lower than the amount usually found in soils that are well stocked with available nitrogen.

The humus results of the different analysts do not agree as closely as they should. This is partially due to an imperfection in the method, which can undoubtedly be remedied—that is, obtaining a clear filtrate with the ammonia treatment. If a clear filtrate is not obtained, hydrated clay-like particles are present in the humus residue when it is ignited, which gives too high results. I do not think the humus methods have been tried by a sufficient number of analysts to entitle them to a place as official methods. The results are encouraging, and with improvements in the methods, and their application to a greater variety of soils known to be deficient, and well supplied with available nitrogen, a safer conclusion can be reached regarding their value.

It may also be added that the results on total nitrogen agree very well, and seem to indicate that the association method for that determination is now about all that could be desired.

Undoubtedly one of the most important features of the work this year has been that on the availability of phosphoric acid as determined by treatment with N/5 HCl. The difference between the two soils has been brought out in a very striking manner by this solvent. In every case the results agree with the known facts in saying that sample No. 2 has a much larger amount of available phosphoric acid than No. 1. With one or two exceptions, the results by the different analysts agree very well—quite as well in fact as could be expected with a new method.

Sample No. 2 having received 350 pounds of superphosphate per annum for more than fifty years, would naturally be expected to contain considerably more available phosphoric acid than No. 1, as the amount applied has been greatly in excess of the amount removed by the crops. There has, in fact, been so much added that the difference is seen in the totals. In every case a larger amount of total phosphoric acid has been found in No. 2 than in No. 1. It will be observed, however, that the dilute acid very much magnified the difference in phosphoric acid content between the two soils. While the ratio between the average totals from the two soils is 1 to 1.55, the corresponding ratio from the N/5 acid is 1 to 9.16.

As a further check on the N/5 HCl method, through the kindness of Mr. Wiley and Sir J. H. Gilbert, samples from a number of the Rothamsted Broadbalk wheat-field plots were secured, and both the total phosphoric acid and that soluble in N/5 HCl determined.

The results secured are given in Table No. 3.

TABLE NO. 3.—*Results calculated water free.*

[Rothamsted soil samples—Broadbalk wheat field.]

No phosphates added for 53 years.					Fertilized with 350 pounds superphosphate per annum for 53 years.				
Gilbert mark.	Reaction with litmus paper.	Per cent moisture (five hours).	Per cent total P_2O_5 (Goss method).	Per cent P_2O_5 with N/5 HCl.	Gilbert mark.	Reaction with litmus paper.	Per cent moisture (five hours).	Per cent total P_2O_5 (Goss method).	Per cent P_2O_5 with N/5 HCl.
W3	Alkaline	1.59	0.11	0.005	B4A	Alkaline	1.63	0.19	0.034
B3A	Alkaline	1.43	.11	.003	B2A	Alkaline	1.87	.17	.037
B1A	Alkaline	1.81	.11	.002	W11	Alkaline	1.77	.18	.043
W10A	Alkaline	1.75	.10	.004	W13	Alkaline	1.82	.19	.045
Av		1.65	.108	.0035	Av		1.77	.183	.0398

By referring to the table it will be seen that the analytical results check with the known facts in every instance. While there is, in every case, a difference in amount of total phosphoric acid between the plats receiving phosphatic fertilizers and those not receiving the same, this difference is greatly magnified in the results secured with the N/5 acid. The ratio between the average totals is 1 to 1.7, while the corresponding ratio for the N/5 HCl is 1 to 11.4.

Through the kindness of Messrs. Frear and Armsby, two composite samples from the Pennsylvania Experiment Station fertilizer plats were also secured. These samples were analyzed in the same way as the Rothamsted samples recorded in Table No. 3. The results secured are given in Table No. 4.

TABLE NO. 4.—*Results calculated water free.*

[Pennsylvania Experiment Station. Soil samples from Tier I. general fertilizer series.]

Sample I.—From plats 1, 8, 14, 24, 36. No fertilizer added for 16 years.				Sample II.—From plats 11, 21. A total of 384 pounds phosphoric acid per acre added in 16 years.			
Reaction with litmus paper.	Per cent moisture (five hours).	Per cent total P_2O_5 (Goss method).	Per cent P_2O_5 with N/5 HCl.	Reaction with litmus paper.	Per cent moisture (five hours).	Per cent total P_2O_5 (Goss method).	Per cent P_2O_5 with N/5 HCl.
Neutral	1.12	0.09	0.0046	Neutral	1.35	0.13	0.0084

Dr. Frear states that the unfertilized plats have been proved deficient in available phosphoric acid, as adjacent plats have responded quite freely to the application of phosphates. By referring to the table, it will be seen that but a small amount of phosphoric acid was secured from the sample from the unfertilized plats, an amount about the same as that secured from the Rothamsted plats receiving no phosphates, while nearly twice as much was obtained from the plats which had received 384 pounds of phosphoric acid per acre in sixteen years. This, it appears to me, is again in accordance with the facts in the case. It will be noted that only a comparatively small amount of phosphoric acid was added to the fertilized plats, an amount equal to but 24 pounds per acre per annum. It is no doubt true that a considerable portion of this was removed in the increased crops secured by the use of the complete fertilizer employed, so that one would not expect a very great increase of available phosphoric acid in the sixteen years that the experiments have been in progress.

The above evidence, added to that secured last year, would, it seems to me, justify us in strongly recommending the N/5 HCl method for phosphoric acid for adoption as an official method of the association.

There are one or two points in the method as it stands at present, however, that should be changed before it is adopted.

Mr. Peter has called attention to the fact that all of us who have worked with the samples this year have doubtless observed that the final precipitate from the soil deficient in phosphoric acid is extremely small. This necessitates exceptionally careful work to avoid error. While this precipitate is still many times larger than the gold bead secured in the ordinary gold assay, still I agree with Mr. Peter in thinking that it would be conducive to more uniform results to increase the size of the precipitate by increasing the amount of the soil taken in the case of soils thought to be low in available phosphoric acid, and shall therefore make that recommendation.

It has been thought by one or two that the short time allowed in the method for the formation of the yellow precipitate is not sufficient, in the case of such small amounts of phosphoric acid as are present in soils deficient in that substance. While the referee very strongly believes the contrary, based on results from a large number of experiments, conducted for the purpose of settling that point, a considerable number of which has been reported in the A. O. A. C. proceedings in the past, still, as it can do little harm for the yellow precipitate to stand longer than ten minutes at 80°, it might be well to make the directions read: Allow to stand not less than ten minutes after removing from the 80° bath. This will permit those who have doubts on the above point to allow the precipitation to proceed over night if desired.

In the preliminary digestion to determine the amount of acid neutralized by the soil, it is quite unnecessary to take as much as 20 grams of soil and 200 cc of acid. Half these amounts is ample.

Mr. Moore, of the United States Department of Agriculture, seems to have mistaken the intention of the referee in that instead of increasing the strength of the N/5 acid sufficiently to allow for the acid neutralized by the soil and keeping the volume the same, he secured the increased amount of acid by taking a larger volume of N/5 acid. This point should perhaps be a little more definitely brought out in the directions. It seems to the referee to be more conducive to concordant results to use the same volume of solution in each case.

By having a large stock solution of normal or half normal HCl on hand, it is very little trouble to calculate the number of cubic centimeters of the same and the number of cubic centimeters of water necessary to be used to make a liter of acid of the desired strength. For the preliminary digestion all that is necessary is the N/2HCl used by many in the nitrogen work, which same can be very readily diluted to make N/5 acid.

Mr. Peter did some valuable experimenting on the samples this year other than that called for in the directions. This work will be found recorded in Tables Nos. 1 and 2. Among other things he showed that N/5 nitric acid gave results quite as good on these soils as N/5 HCl, and in some ways he thought it might be preferable to HCl.

In regard to the other determinations made in the soil samples, perhaps all the information that is necessary concerning them can be secured by referring to Tables Nos. 1 and 2.

THE ASH SAMPLE.

The results secured on the ash sample are given in Table No. 5.

TABLE NO. 5.—*Ash of tornillo wood (Prosopis pubescens).*

Substances.	J. K. Haywood, U. S. Dept. of Agr.	G. L. Teller, Arkansas.	A. M. Holt, New Mexico.	L. M. Bloomfield, Ohio.	Average.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Soluble SiO_2	11.12	10.73	.....	7.37	9.74
Insoluble SiO_2 (sand)	10.29	11.16	.....	14.70	12.05
Total SiO_2	21.41	21.89	21.40	22.07	21.69
K_2O	11.05	11.03	11.03	11.35	11.11
Na_2O	3.57	3.87	4.32	6.89	4.66
CaO	35.55	35.10	34.57	34.46	34.92
MgO	8.97	7.18	6.92	8.12	7.80
Mn_3O_4	.24	.23	.27	.48	.30
Fe_2O_3	.....	1.00	.....	.....	.....
Al_2O_3	.....	2.28	.....	.....	.....
$Fe_2O_3Al_2O_3$	1.35	(3.28)	3.32	.97	2.23
P_2O_5	3.28	3.39	3.46	3.45	3.40
SO_3	.67	.66	.87	.59	.70
CO_2	12.75	12.28	12.49	.....	12.51
Cl	.26	.60	.22	.23	.33
C	.78	.67	1.51	.88	.96
Moisture	.22	.....	.18	.....	.20
Total	100.10	100.18	100.56	.....	.....
O equivalent of Cl	.06	.13	.05	.....	.....
Corrected total	100.04	100.05	100.51	.....	.....

It will be seen by referring to the table that the agreement of results on several of the determinations is very good. There are other determinations, however, on which the agreement is not so good as could be wished. This is particularly true in the case of soluble and insoluble silica, iron, aluminum, and soda.

As the methods for the proposed separation of soluble and insoluble silica have been shown to be utterly unreliable (see Proceedings of the Eleventh Convention of the A. O. A. C., pp. 43, 44), and as this separation is, to say the least, of very doubtful utility, it would seem to be well to combine the soluble and insoluble silica determinations in one of total silica. This would shorten the work and result in more uniform results. This, then, will be offered as a recommendation.

The way the method for potash and soda stands at present, if literally followed, may not result in a complete separation of the alkali chlorids from lime, etc. The referee believes that both ammonium carbonate and oxalate should be used in precipitating out the lime, etc., and that the process of purification will in some cases have to be carried even further than called for in the directions. This will constitute another recommendation.

In the New Mexico laboratory it has been found impossible to secure reliable results in the determination of chlorin by the volumetric method, notwithstanding the fact that we constantly employ that method in the analysis of waters, and should be familiar with the same. The end reaction in the case of ash solutions is frequently so indistinct that the exact point can not be told with any degree of certainty. On the other hand, the gravimetric method has been found to give perfectly reliable results. The referee therefore thinks that the volumetric method for chlorin in ash should be stricken out and the gravimetric method required.

Recommendations.

The referee this year wishes to leave as clear a field as possible for his successor, and has therefore only one recommendation to make in regard to the work that shall be taken up next year, and that is:

(1) That the ammonium and calcium chlorid methods be tried again next year on the Rothamsted samples Nos. 16069, Gilbert mark W 10A, and 16070, Gilbert mark W 13, if Mr. Wiley have sufficient of the same. No. 16069 has been in wheat continuously for fifty-three years and fertilized annually with ammonium salts equivalent to 86 pounds of nitrogen per acre. No. 16070 has been in wheat continuously for the same length of time and fertilized annually with 400 pounds of ammonium salts, 350 pounds superphosphate, and 200 pounds sulphate of potash.

(2) It is recommended that the N/5 HCl method for available phosphoric acid, in the form given below, be adopted as an official method of the association.

Determination of phosphoric acid soluble in N/5 HCl.

(*a*) Prepare a large stock solution of normal HCl by titrating against a standard KOH solution containing little or no carbonate, using phenolphthalein as the indicator.

(*b*) Digest 10 grams of air-dried soil, in a stoppered flask, with 100 cc of N/5 HCl, for exactly five hours in a water bath kept at a temperature of 40° C. Filter off the solution through a dry paper, cool to the room temperature, and titrate 20 cc of the filtrate with standard carbonate-free KOH solution, using phenolphthalein as the indicator. From the data thus secured, calculate the exact number of cubic centimeters of normal acid from the stock solution and of water to combine to make exactly one or two liters of acid of N/5 strength after allowing for the amount neutralized by the amount of soil to be taken in (*c*).

(*c*) Transfer 200 grams of the air-dried soil to a large, dry, glass-stoppered bottle or other similar vessel, and add exactly 2,000 cc of N/5 HCl corrected for neutralization as in (*b*). (In the case of soils known to be rich in available phosphoric acid 100 grams of soil and 1,000 cc of acid will be sufficient.) Place the bottle in a large water bath (a metal washtub is excellent) and keep at a temperature of 40° C. for exactly five hours, shaking thoroughly each half hour. At the end of the digestion shake contents of bottle well and pour quickly upon a large, dry, ribbed filter of two thicknesses of paper and of sufficient size to receive the entire contents of the bottle. The filtrate is to be received in a dry vessel and the solution poured back through the paper until entirely free from soil particles. Evaporate 1,000 cc of the filtrate if 200 grams of soil were taken, or 500 cc if 100 grams were taken, to dryness in a porcelain dish, after adding a few cubic centimeters of nitric acid to oxidize organic matter, etc., moisten the residue with HCl, take up with water, and filter into a flask of about 500 cc capacity. Add 15 grams of ammonium nitrate in solution, add strong ammonia until a permanent precipitate forms, and then concentrated nitric acid until the precipitate dissolves. Dilute the solution to about 100 cc, if not already to that volume, place a thermometer in the flask, and heat to exactly 85° C. Add 75 cc of recently prepared molybdate solution, digest in a water bath at 80° for fifteen minutes, with occasional shakings, remove from the bath and allow to stand at least ten minutes before filtering. Continue the determination as given in Bulletin No. 43 of the Division of Chemistry, United States Department of Agriculture, pages 58 to 60.

(3) It is recommended that in the third line from the bottom, first paragraph under "Analysis of the ash," Bulletin No. 46, Chemical Division, United States Department of Agriculture, page 44, the words "an S. & S. hardened filter" be stricken out and the words "a tared Gooch crucible" be substituted instead. Further, that section (*a*) be changed to read as follows:

(*a*) *Determination of carbon and silica.*—The carbon and silica in the Gooch crucible are to be dried at 120° C. to constant weight, incinerated to constant weight, and the amount of carbon determined from the loss in weight.

(4) That on page 45, in the fourth and seventh lines from the top, the words "and ammonium oxalate" be inserted after "ammonium carbonate," and that at the end of the paragraph the following be added: "Test the purity of the chlorids by dissolving same in a few cubic centimeters of water and adding a few drops of ammonium

carbonate and oxalate solution. If a precipitate occur, or if there be an insoluble residue upon dissolving in water the same must be filtered off, the solution evaporated to dryness, ignited, and reweighed. In any case the ammonium salts must, of course, be ignited off before the potash determination is made."

(5) That section (*f*) be changed to read as follows:

(*f*) *Determination of Chlorin.*—Boil 10 grams of the ash in 400 cc of pure water for half an hour, transfer to a measuring flask of 500 cc capacity; cool, make up to volume, filter off through a dry folded paper, transfer 100 cc of the filtrate to a beaker, acidify with nitric acid, heat to boiling, add silver nitrate solution in excess, and stir vigorously until the precipitate collects. Filter off the precipitate through a tared Gooch crucible, wash thoroughly with hot water, dry at 110°, and weigh the AgCl secured.

CONCLUSION.

In conclusion the referee wishes to thank those who have taken part in the work this year for their cooperation, and to state that he believes the association will agree in saying that the subject of soil analysis is another step forward as a result of the year's work. To his successor he would say, Let the good work proceed.

Mr. Wiley read a letter from Mr. Caldwell, expressing regret at not being able to be present. He also read a letter from the referee on sugar, stating that owing to illness in his family he could neither be present nor prepare a report.

REMARKS OF SECRETARY WILSON.

The Chair at this point introduced the honorable Secretary of Agriculture, who had just entered the hall. The Secretary was greeted with applause. He said:

MR. PRESIDENT AND GENTLEMEN: I have merely stopped in to add to your welcome to the capital city of the nation. I am glad to see you. As a representative of the farming classes of the country, I am proud to say in your presence that we recognize fully the necessity of the chemist in all advances that are being made along lines of progress in agriculture. I need only mention briefly the work being done all along the line.

In dairy matters we have Babcock and Patrick, who have helped the farmer to better methods and enabled the American farmer to put his products on the markets of the world and compete successfully with dairy production of the famed countries of Europe. The chemist has helped us to do this. I might mention the work being done along the line of making the United States independent of other countries in sugar. The work being done by many of our experiment stations along this line is of great value. A hundred million dollars is hung up within reach of the American farmer, and the chemist is telling him how to get it, analyzing his beets, and telling him what soils are best for his purposes. Permit me to assure you that the retention of this hundred million dollars among our people will be due in great part to the excellent work being done by the chemist. I mention these things only to show the appreciation of your work by those who till the soil. As regards the light in which we estimate you here, I stand ready to

make good whatever Dr. Wiley wants done by the Department of Agriculture. (Applause.)

I have but one ambition here that I very greatly desire to have gratified during my short stay in connection with the Department of Agriculture. If I can succeed, through the help of the corps of scientists connected with the Department, in impressing future Presidents of the United States with the fact that no man should be Secretary of Agriculture without a scientific knowledge of agriculture, then my ambition will have been satisfied. No matter what the political complexion of the President of the United States who may succeed the present occupant of the White House, I would like to have him select for Secretary of Agriculture a man who understands not only the practical but the scientific side of agriculture. I think this may possibly be done if we succeed fairly well in impressing the country that the work we are doing is good, honest, helpful work now.

I thank you for your attention, and will detain you no longer. It is a serious thing to interrupt scientific gentlemen, even for a moment. (Applause.)

The Chair expressed the gratification of the association at the Secretary's remarks.

Mr. Wiley read a letter from Professor Hilgard, calling attention to a paper, on the objects and methods of soil analysis, read at the Minneapolis meeting of the Association of Agricultural Colleges and Experiment Stations:

THE OBJECTS AND METHODS OF SOIL ANALYSIS.

Paper of E. W. Hilgard, read before the Detroit meeting of the Society for the Promotion of Agricultural Science.

Among the recommendations made to the Association of Official Agricultural Chemists by the referee for 1896 (Professor Goss) the first reads as follows:

"That the complete analysis of a strong acid solution of the soil, as given last year and in preceding years, be stricken out of the official methods of the association."

In the referee's record of work preceding these recommendations he shows a keen insight and presents so many new and important facts regarding the action of root solvents upon soils and the variety of causes that may influence their action that I can not but express surprise and regret at the recommendation above quoted. It appears to me to tend in the direction of narrowing down the function of the agricultural chemist to that of a mere analyst, dependent upon figures alone—the very state of things which has contributed most to the discredit so long attaching to chemical soil analysis. As the same referee has been reappointed for 1897, and will be sure to carry out the recommendations as adopted with the same ability manifested heretofore, I desire to interpose a demurrer against the argument upon which he bases the above recommendation.

Especially on the ground that potash, phosphoric acid, and nitrogen are the only three elements needed to be supplied in fertilizers, Goss argues that these are the only ones required to be determined by the analyst as to their total quantity and availability. "Practical analysis of the soil," he says, "should tell us with reasonable certainty the amount and availability of these three substances as present in the soil, and then we will have accomplished the principal thing that is at present

demanded of the soil analyst." He admits that it is interesting, from a scientific point of view, to know something of the other ingredients, and that they may have some bearing on plant production; but thinks that the time required for their determination could be better used for other purposes.

But what does Goss mean by "practical analysis?" The word "practical" has been much abused in connection with agricultural science and practice. It is launched against us as a kind of thunderbolt, both by the men who wholly disbelieve in the application of science to agriculture, as well as by the politician, who tries to make capital by denouncing our agricultural experiment stations for pursuing any line of research which does not appear to him to bear directly upon the operations of the farm. Now, if in "practical analysis" Goss speaks only with reference to satisfying the demands of the public, I must dissent from him as to the propriety of attempting to conciliate these two classes at the expense of the true objects and methods of investigation, for which our stations were established. Such concessions are not only directly harmful to the true purposes of station work, but indirectly lead only to further inadmissible demands on the part of the self-styled practical man.

While, as I have always insisted, the stations ought assuredly first to cater to the pressing needs of the population by their work, yet they will be untrue to their mission if they fail to develop at the same time agricultural science from its scientific side, and thereby aid in enabling it to enlarge its functions beyond the mere prediction of the present productive capacity of land and its immediate needs in fertilization. But even as regards fertilization, it is quite obvious that the kind of fertilizers to be used should depend in very many cases essentially upon the presence or absence of other ingredients than the three furnished in the fertilizers. From the latter point of view, Goss's recommendation is not well advised, even if only meant to apply to the commercial chemist who does not go beyond the supplying of figures to his client, leaving the latter to interpret them for himself as best he may. It is not enough that his figures should show that nitrogen, phosphoric acid, or potash are deficient or abundant, but it must also be shown what, from the general character of the soil, is the *form* in which these substances should be supplied, and in what manner. The chemist should, for instance, be able to tell the farmer whether his soil is such that the application of a superphosphate to the surface will, in the ordinary course of events, assure a supply of that substance to the roots of his crops; or whether, e. g., the presence of an excess of calcic carbonate will prevent the penetration of the superphosphate to the depth of not more than an inch or so; or whether, as in the highly ferruginous soils of Hawaii, any water-soluble phosphoric acid will, in a short time, be definitely locked up in the insoluble form, so that, instead of a superphosphate, it should be applied in the form of phosphate slag, or in that of di-calcic phosphate. Speaking quite generally, the presence or absence of lime is, and has long been known to be, such a potent determining factor in the producing qualities of soils, that the proposition to omit its determination as a part of the regular analysis for practical purposes will sound strangely in the ears of agricultural chemists the world over. It means that it is of no practical consequence to know whether a soil is naturally marled already or requires artificial marling to improve its chemical or physical condition, or both; whether it is naturally adapted to the production of leguminous crops, and a score of other points which are everywhere considered of importance to the farmer.

Similarly in the case of potash, it is not a matter of indifference whether or not a large proportion of zeolotic silicates is present, by which it may be retained, or whether the next rain may wash it through the soil beyond the reach of the roots. From this point of view alone, then, the determination of lime, iron, and "soluble" silica are of direct importance, even according to the present imperfect state of our knowledge. But how shall that knowledge ever be perfected if the recommendation of the referee as to the determination of the three ingredients alone should be accepted by our experiment station workers? It seems to me that the

very fact quoted by Goss that "we know but little about the relation of these substances to vegetable nutrition" should lead him to reverse his conclusions, at least so far as the experiment station work is concerned.

The recommendation works the wrong way also in the case of virgin soils; for in the case of cultivated land something at least is usually known in a general way regarding the character of the soil and the main points to be observed in their treatment. As I have frequently urged, in virgin soils the proportion of actually available plant food may ordinarily be presumed to be proportionate to the total amounts present as ascertained by the usual methods of analysis; so that the determination of actually available plant food by the method of Dyer or others becomes for the time being unnecessary. But it is of the greatest importance to know whether such soils are, for instance, sufficiently or highly calcareous or not, since that indicates from the outset not only their adaptability or nonadaptability to certain important cultures, but also the sufficiency of their potash supply. In the case of ferruginous soils again, it is of the highest importance to inform the farmer that if he allow the subsoil to become swamped by an overuse of irrigation water or otherwise, that subsoil will become not only useless but positively poisonous, not only to roots within it, but also to those some distance above it, because of the reduction of ferric hydrate to ferrous carbonate, or other ferrous salts. In irrigation regions it is often of paramount importance that this should be known beforehand.

I contend, therefore, that the determination of lime, ferric oxid, and soluble silica is of such general importance, not only with a view to the general qualities of the soil under cultivation, but also with reference to the use of fertilizers, that to omit these determinations and to restrict soil analysis to the "practical" determination of potash, phosphoric acid, and nitrogen, is inadmissible as tending to lead to erroneous inferences, not only theoretically, but practically.

But if these determinations be made, it is difficult to see wherein the process of general analysis as heretofore practiced can be materially abridged. It is true that we might omit the determination of the alumina, determining only the iron by titration of the solution of the joint precipitate. But this is a very trifling abridgment and prevents the comparison of the amount of soluble silica with that of alumina with reference to kaolinite, which, as I have heretofore shown, frequently goes to prove the presence of free aluminum hydroxid in the soil. It is true that we do not know the exact significance of this substance in connection with the soil functions, but it is certainly desirable that we should obtain this knowledge hereafter; and to omit this determination for the sake of so slight a saving of time seems to me to be a deviation from the principles of sound investigation, of which certainly the stations should not furnish the example.

As regards other substances, viz, magnesium and manganese, I agree that manganese might very well be let go in with iron, if this were compatible with the proper separation from the lime; for, so far as we know, manganese exerts little or no influence upon vegetable nutrition, and its functions in the soil appear to be of no special significance. But it is quite otherwise with magnesia. It is of the utmost importance, next, probably, to lime; its functions in the soil may or may not be restricted to the neutralization of acid, and therefore the prevention of acidity, and the formation of the zeolitic silicates. But so long as it is omnipresent—and frequently in very large proportions—it seems highly expedient, from the standpoint of general scientific investigation, to make its determination an invariable practice. We have no method of separating potash from it without so close an approach to its actual gravimetric determination that, as in the case of alumina, it seems almost wantonness to omit to weigh it for the sake of so slight a saving of time. If we were to adopt the method of Grandeau, which precipitates potash by perchloric acid from a complex solution, there would be better reason for urging the abandonment of the determination of magnesia. Again, if the abridgment of labor be the point so essentially desired, I am at a loss to see the propriety of substituting in the determination of the total phosphoric acid the sulphuric acid method of Goss for the one with nitric acid, as heretofore practiced. There is no question that the sulphuric-

acid method requires much more time and attention on the part of the operator, and it is liable to a number of accidents from which the nitric-acid method is entirely free. The only claim it can make for consideration is that it extracts the phosphoric acid more completely in case of the presence of highly insoluble compounds, such as crystallized vivanite and other phosphate minerals. But it may be seriously questioned whether the presence of such minerals in the soil is of any "practical" use whatever. I question that it is even of theoretical interest to the investigator of soils from the agricultural standpoint, however interesting from that of the geologist or mineralogist. As in the case of the use of sulphuric acid for the determination of alumina in soils, it leads to an overestimate of the soil's capacity to supply the ingredient sought. It stands on a similar plane of utility as the determination of potash by means of hydrofluoric acid, which, so far as I can see, is totally devoid of interest from an agricultural standpoint. It is not reasonably to be supposed that any organic acid, not even oxalic, to the dilution in which it is applied by the plant is likely within any time in which we are interested to go beyond the action of strong hot nitric acid. I do not think that Goss has presented any reasons for abandoning the nitric acid digestion as a method of determining the phosphoric acid in soils sufficient to justify the substitution therefor of his very thorough and ingenious but troublesome and excessively incisive method. When I find in Hawaiian soils that yield to the Dyer solvent only 0.004 of P_2O_5 by the nitric-acid method nearly 1 per cent, it is certainly of no practical interest to me to know that HCl will take out of the same soil nearly twice the amount that nitric acid did. Nor will it be of any practical interest to obtain still more by means of the Goss sulphuric-acid method.

The most important step in advance shown by Goss's report, and for which if confirmed by further experience the agricultural world will be grateful to him, is the substitution of dilute HCl for the determination of available potash and phosphoric acid instead of the organic acids heretofore used. While I must say that within my own experience thus far the citric-acid solution has given excellent results whenever the neutralization of carbonate of lime and magnesia in the soil has previously been properly observed so as to render the solvent of uniform strength, yet the much greater facility with which HCl may be used in analytical operations renders its use so desirable that it must be earnestly hoped it will prove as much of a success as now seems probable. The remarkable effect of calcium chlorid, however, causes me to suggest that the effect of neutralization would in the case of HCl be quite different from that of citric acid so far as potash is concerned; for in many cases the amount of calcium chlorid formed by neutralization will greatly exceed in its effects that of the HCl itself, and it would therefore seem that as a general reagent for the determination of available potash the latter is unavailable for the great majority of soils.

The convention then adjourned until 2 p. m.

WEDNESDAY—AFTERNOON SESSION.

ELECTION OF OFFICERS.

The committee on nominations presented the following report:

The committee on nominations begs to present the following names for the several elective offices of the association:

For president, Mr. A. L. Winton, of Connecticut; for vice-president, Mr. R. C. Kedzie, of Michigan; for secretary, Mr. H. W. Wiley, of Washington, D. C.; for members of the executive committee, Mr. M. A. Scovell, of Kentucky, and Mr. J. L. Hills, of Vermont.

C. L. Penny,
L. L. Van Slyke,
Elton Fulmer,
Committee on Nominations.

Upon motion the report was accepted.

Mr. Bigelow moved that the Chair be instructed to cast the ballot of the association for the officers nominated. Carried, and the officers named declared duly elected.

VISIT TO DEPARTMENT OF AGRICULTURE.

The PRESIDENT. The regular order of business is to proceed to the Department of Agriculture, as arranged. Before doing so, however, I desire to name the committee to confer with the committee from the Association of Agricultural Colleges and Experiment Stations on the subject of fertilizers. In the appointments I have tried to secure as wide representation of the different sections of the country as possible, and name as the committee to represent this association Messrs. Wiley, of Washington, D. C.; Kilgore, of Mississippi; Huston, of Indiana; McDonnell, of Maryland, and Ross, of Alabama.

The convention thereupon adjourned to visit the Department of Agriculture.

The members of the association visited first the chemical laboratory, where they inspected the methods of sugar-beet analysis and the polarimetric room. The methods of mounting and adjusting the standard instruments and of illuminating their scales were studied. The operation of the bomb calorimeter for the combustion of bodies in a compressed atmosphere of oxygen was illustrated. The members then proceeded to the office of the Secretary of Agriculture and were most cordially received by the Secretary in his office.

THIRD DAY.

THURSDAY—MORNING SESSION.

Called to order at 9.30. No member of the committee on recommendations being present, the regular order was postponed and the Chair called for the report on foods and feeding stuffs.

Mr. Lindsey read the report, as follows:

REPORT ON CATTLE FEEDS.

By J. B. Lindsey.

I.

Estimation of Starch.

Among the so-called standard methods for the estimation of starch may be mentioned those of Sachsse, Märcker, Reinke, Asboth, Guichard, Baudry, and the diastase methods. Most of these methods give fairly concordant results when the substance examined contains no other carbohydrates than pure starch. Stone[1] has shown, however, that when the most of these methods are employed on materials containing a variety of carbohydrates, very different starch percentages result. Sachsse's method, which consists in boiling the substance directly with dilute hydrochloric acid, is recommended by the Association of Official Agricultural Chemists for use only in estimating the starch content of commercial starch and potatoes. The other methods which have seemed best adapted for the estimation of starch in agricultural products, are the diastase and the Märcker methods; also the Märcker method as modified by Reinke.

A year ago your referee compared the diastase and Märcker methods without securing any very decisive results, consequently both were adopted by the A. O. A. C. as provisional methods until further investigations could be made.

The work on starch this year consisted in comparing the diastase, Märcker, and salicylic-acid or Baudry modified methods on three different substances. Our principal object in testing the Märcker method was to note whether heating for one-half hour, under 3 atmospheres in one-tenth of 1 per cent tartaric-acid solution, would exert any solvent action on carbohydrates other than starch. The Reinke modification of the Märcker method consists in omitting the diastase treatment, using one-half of 1 per cent lactic acid, and in heating for two and one-half hours under 3½ atmospheres. We preferred to test the original Märcker method, feeling sure that the Reinke modification would have a greater solvent action than the method originally brought forward by Märcker. The methods as sent out were described as follows:

I. diastase method.

Exhaust 3 to 5 grams of substance with ether, and treat the residue in a beaker with 50 cc of 10 per cent alcohol for one-half hour with frequent stirrings, filter, and wash with strong alcohol. Bring the dried residue into a beaker with 50 cc of water, immerse beaker in a boiling-water bath, stirring contents constantly until

[1] Journal of the American Chemical Society, **16**, 726.

all the starch gelatinizes; cool to 55° C., add 20 cc of malt extract, and maintain at this temperature until the solution no longer gives the starch reaction with iodin. Filter into a 10-ounce flask, wash until the filtrate amounts to 200 cc, add 20 cc of 25 per cent hydrochloric acid (sp. gr. 1.125), connect with a reflux condenser and heat in a boiling-water bath for two hours, cool, and when lukewarm neutralize carefully with Na_2CO_3 (avoiding excess), and make up to 500 cc. Mix well, pour through a dry filter, and determine dextrose in an aliquot part by Allihn's method. Convert dextrose into starch by the factor 0.9.

II. MÄRCKER'S METHOD.

Free 3 to 5 grams of substance from fat and water soluble material by use of ether and 10 per cent alcohol, as described under diastase method. Dry the residue, powder, and bring into a Linter pressure bottle (if Soxhlet digester is not at hand) with 50 cc of water; immerse bottle in a boiling-water bath until the starch gelatinizes, stirring constantly, cool to 55° C.; add 10 cc of malt extract and maintain at this temperature for one hour. Now add 5 cc of a 1 per cent tartaric acid solution, and heat at 140° C. for one-half hour (or in autoclave at 3 atmospheres one-half hour), or 110° C. for one hour; cool to 55° C.; add 10 cc of malt extract, and maintain at this temperature until the solution no longer gives the starch reaction with iodin. Filter through linen, using suction, into a 10-ounce flask, and wash with water until the filtrate amounts to 200 cc, then add 20 cc of 25 per cent hydrochloric acid and proceed as in the diastase method.

Preparation of malt extract.

Digest 10 grams of fresh, finely ground malt over night at laboratory temperature with 200 cc of water, and filter. Determine the amount of dextrose in a given quantity of the filtrate by boiling with acid, etc., as in the determination of starch.

III. BAUDRY MODIFIED OR SALICYLIC ACID METHOD.

Free 3 to 5 grams of substance from fat and water soluble material as already described. Bring the dried and pulverized residue into a beaker with 100 cc of water, in which is dissolved ½ gram salicylic acid, and heat the beaker in a boiling water bath with constant stirring, until the starch is rendered soluble (10 to 15 minutes). Now filter quickly through linen, using suction, wash with hot water to the volume of 200 cc, add 20 cc of 25 per cent hydrochloric acid, and proceed as under the diastase method.

Some time after the samples necessary for testing these three methods were sent out, Mr. H. W. Wiley kindly sent, at our request, a description of a method employed in his laboratory for the estimation of starch. We compared this with the other three methods.

IV. WILEY'S METHOD.

Extract the fat from 3 grams of the material, dry the residue and digest for two and one-half hours, at 3½ atmospheres, in an autoclave, with ½ gram salicylic acid and 50 cc of water. After cooling, make up to 250 cc, filter, and invert 200 cc of the filtrate in a 500 cc flask with 15 cc of hydrochloric acid (sp. gr. 1.125) for two and one-half hours at 100° C. Neutralize exactly with sodium carbonate, cool, make up to 500 cc, and determine the dextrose in 25 cc by Allihn's method.

The three samples sent out were corn meal, wheat middlings, and lupine seeds, each ground sufficiently fine to pass a ½ mm sieve. The corn meal contained no other known carbohydrate than starch, excepting small amounts of cellulose. The wheat middlings contained, in addition to a moderate amount of starch, liberal quantities of pentosans. The lupine was known to contain pentosans and galactan—both hemicelluloses—in addition to very small or questionable amounts of starch. The presence of starch alone in one substance, of starch and pentosans in a second, and of starch, pentosans, and galactan in a third, would certainly present

conditions suitable for testing the accuracy of the three methods. We present first the results obtained in our own laboratory.

TABLE I.—*Results with different methods of estimating starch.*

Methods.	Per cent of starch in—		
	Corn meal.	Wheat middlings.	Lupine seed.
Diastase	62.53	19.38	2.11
Märcker	65.54	26.84	14.68
Salicylic	57.02	19.03	6.21
Wiley	68.04	34.00	16.41

It will be observed that both the Märcker and Wiley methods yield higher results than the diastase method. This is exceptionally noticeable in the case of the wheat middlings and lupine, which contain other carbohydrates than starch. The salicylic method gave lower results than the diastase in case of the corn meal, about the same for wheat middlings, and higher for the lupine. We are naturally led to inquire the cause of these differences. It would seem at least possible that one or both of the hemicelluloses had been attacked by the action of the several atmospheric pressures. The data throwing light on this point will be found in Tables II and III.

TABLE II.—*Total pentosans, and pentosans present after starch extractions.*

Substance.	Corn meal.	Wheat middlings.	Lupine seeds.
	Per cent.	*Per cent.*	*Per cent.*
Total pentosans	6.02	20.51	9.94
Pentosans after dilute alcohol treatment	5.69	18.30	9.73
Pentosans after dilute alcohol and diastase treatment	4.97	17.14	8.75
Pentosans after dilute alcohol and Märcker treatment	3.70	12.04	5.58
Pentosans after dilute alcohol and Wiley treatment	.29	1.26	1.38

We first determined the total pentosans in each of the three substances, and also after these substances had been treated with the dilute alcohol solution for the removal of water-soluble carbohydrates. A slight decrease in the pentosan percentages is noted as a result of this preliminary treatment. After corn meal had been subjected to diastase treatment for the removal of the starch, the percentage of pentosans decreased from 5.69 to 4.97, or a percentage loss of 12.6. A considerable length of time (four hours) was required to remove all of the starch from the corn. It is possible that the solvent action of the water at 55° C. was in a measure, or entirely, responsible for this decrease rather than the action of the diastase. The Märcker method caused a decrease of from 5.69 to 3.7, or 35 per cent, while in removing the starch by the Wiley method practically all of the pentosans were also removed. The wheat middlings show a percentage pentosan decrease of 6.3 by the diastase, 34.2 by the Märcker, and 93 by the Wiley method. In case of the lupine, a percentage decrease is noted of 10, 42.7, and 85.8 by the diastase, Märcker, and Wiley methods, respectively.

TABLE III.—*Total galactan, and galactan present after starch extraction.*

Substance.	Lupine seeds.
	Per cent.
Total galactan	14.87
Galactan after dilute alcohol treatment	12.25
Galactan after dilute alcohol and diastase treatment	9.36
Galactan after dilute alcohol and Märcker treatment	1.16
Galactan after dilute alcohol and Wiley treatment	0.33

In case of the lupine, which contained a considerable percentage of galactan, the dilute alcohol treatment was found to remove nearly 18 per cent of the entire amount. Of the remaining quantity, the diastase treatment removed 23.7 per cent and the Märcker and Wiley treatments from 90 to 97 per cent. Galactan, in the lupine at least, seems to yield rather more easily than do the pentosans to the action of the various solutions.

We have then positive reasons for the higher starch percentages yielded by the Märcker and Wiley methods above those obtained with the diastase method. It seems established that the several atmospheric pressures render a considerable portion of these hemicelluloses soluble, or possibly even partially destroy them. The Wiley method acts more severely, because of the increased length of time during which the substances are under pressure.

If the Märcker method act thus on the hemicelluloses, it is safe to assume that the Reinke modification would exert a severer action. In fact, Märcker[1] says: "The Reinke method may give perphaps too high results; but, on the other hand, the Märcker method yields positively too low results." He makes emphatic, however, that "the Reinke method is to be used only for those materials which, during the manufacturing process, are subjected to high pressures; it yields at least 3 per cent too high results for all other conditions." The diastase method is now recommended and used by Märcker for the estimation of starch in fodders. The substances causing the high results in case of the pressure methods he does not state, but simply refers to them as "nicht Stärke," i. e., substances not starch. This, at least, leaves one to assume that the pressure acts partially on the cellulose and partially on other carbohydrates not specified.

In view of the facts presented, we feel bound to condemn for the estimation of starch the Märcker, Reinke, and Wiley methods. It seems probable that any method requiring the substance to be subjected to pressure would be included in this condemnation. The fact is not overlooked, however, that in the diastase method either the diastase or the water exercises a slight solvent action on the hemicelluloses, which probably tends to increase the percentage of true starch. In the following table are given the results obtained by different analysts with the diastase, Märcker and salicylic methods:

TABLE IV.—*Results obtained by other chemists.*

Analysts.	1. Corn meal.			2. Lupine seed.			3. Wheat middlings.		
	Diastase method.	Märcker's method.	Salicylic method.	Diastase method.	Märcker's method.	Salicylic method.	Diastase method.	Märcker's method.	Salicylic method.
	Per ct	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
W. H. Krug, U. S. Dept. of Agr.	69.28	71.34	.....	2.58	16.79	.....	[1]30.94	[1]35.58	.....
O. W. Knight, Me. Exp. Sta.....	61.47	.....	52.50	None.	.....	7.51	[1]15.89	.....	19.11
C. D. Howard, N. H. Exp. Sta ...	65.19	.....	.....	2.43	.....	.....	20.46	.....	.....
C. A. Brown, jr., Penn. Exp. Sta.	67.15	.....	62.98	2.40	.....	11.63	21.01	.....	[1]25.00
C. H. Jones, Vt. Exp. Sta	61.17	.....	55.68	1.64	.....	1.21	20.14	.....	17.00
H.C. Sherman, Columbian Univ.	67.79	68.30	67.68	.....	.....	.....	21.00	24.68	22.18
E. B. Holland, Mass. Exp. Sta...	62.53	65.54	57.02	2.11	14.68	6.21	19.38	26.84	19.05
Average	64.94	68.39	59.17	2.23	15.73	.....	20.4	25.76	19.3

[1] Excluded from average.

While wide variations occur in the figures presented by different analysts, they, nevertheless, make emphatic the conclusions drawn from our own work. In the results obtained with corn meal, three analysts secured by the disastase method 61.72 per cent of starch, three others 68.07 per cent, and one 65.19 per cent. It is to

[1] Die Agricultur Chemische Versuchsstation Halle: ihre Einrichtung und Thätigkeit, p. 113.

be confessed that these differences are difficult to explain. The writer endeavored to obtain all of the data necessary for recalculation, but was only partially successful. Very few chemists undertook the Märcker method, not possessing an autoclave. For the corn meal the average results by this method are higher than those obtained by the diastase method, while with the salicylic method the results are lower. In case of the lupine, six analysts employing the diastase method find but very small percentages of starch, while the Märcker method shows 16 per cent. With wheat middlings five chemists employing the diastase method obtained very encouraging results, one too low, and one evidently too high. The Märcker method again yields high figures, while the salicylic acid agrees fairly well with the diastase method.

REMARKS.

The differences in the percentage of starch secured by different analysts may be accounted for as follows:

In the description of the method sent out, it was advised to free the substance from water-soluble material by treatment for one-half hour in a beaker with 50 cc of 10 per cent alcohol, stirring frequently. We found later that this method did not seem to remove all of the soluble material, so, in our case, the substance was brought on a filter and washed with 150 cc of 10 per cent alcohol.

Secondly, some analysts might not have correctly calculated the amount of dextrose to be deducted for the malt solution. In the third place, the methods employed for reducing the copper might have caused variations. It is believed a particular method should have been specified. At first we reduced the copper in hydrogen, but experienced some trouble because the metal, on its removal from the stream of hydrogen, after it had become cool, immediately oxidized on the surface and increased in weight. We, therefore, substituted a stream of dry oxygen, and weighed as copper oxid, with highly satisfactory results. A few comparative tests on a 1 per cent solution of pure dextrose gave results in favor of the oxidation method. We propose to still further investigate this point. Again, the substances should have been ground finer. They were all passed through a one-half mm sieve, but we believe they should have been ground to near a powder, in order to allow the diastase to do thorough work.

The salicylic or modified Baudry method has a tendency to give low results when employed on substances containing high starch percentages, because of the difficulty experienced in getting all of the starch into solution. Fine grinding may, in a measure, overcome this difficulty. Again, it is difficult to know how long the hot salicylic acid solution can be left in contact with the substance, because of its tendency to attack both the pentosans and the galactan. (Note the results obtained with the lupine in Table IV.)

RECOMMENDATION.

We recommend that the diastase method be declared the provisional method of the A.O.A.C. for the estimation of starch in all substances, excepting in commercial starch and in potatoes.

Diastase method.

Extract from 2 to 5 grams of the finely powdered substance with ether, bring the extracted residue onto a filter, or into a Gooch crucible, and wash with 150 cc of 10 per cent alcohol, and then with a little strong alcohol. Place the residue in a beaker with 50 cc of water, immerse the beaker in a boiling water bath, and stir constantly until all of the starch is gelatinized; cool to 55° C.; add from 20 to 40 cc of malt extract and maintain at this temperature until the solution no longer gives the starch reaction without iodin. Cool and make up directly to 250 cc; filter; bring 200 cc of the filtrate into a 10-ounce flask with 20 cc of 25 per cent hydrochloric acid (sp. gr. 1.125); connect with a reflux condenser and heat in a boiling-water bath for two hours; exactly neutralize while hot with sodium carbonate, avoiding excess, and make up to 500 cc. Mix the solution well, pour through a dry filter, and determine the dextrose in an aliquot part by Allihn's method, pages 53–57, No. 46. Convert the dextrose into starch by the factor 0.90.

Percentages of Pentosans and Galactan in Ordinary Cattle Feeds.

We desire at this point to call the particular attention of chemists to the very general distribution of the pentosans and to the extent of the galactan in agricultural plants and seeds. If methods where pressure is employed for the determination of starch do render soluble a considerable portion of those other carbohydrates, it is evident that their value must be exceedingly limited. The percentages of pentosans and galactan in various feeds are indicated in the tables given below:

Table V—*Pentosans.*

[Partly our own work, partly compiled.]

Cattle feeds.	Per cent.	Cattle feeds.	Per cent.
Hay of various grasses	15–22	Wheat meal	8.78
Hay of salt grasses	24–28	Soy bean meal	5.12
Rye straw	24.84	Wheat bran	15–29
Wheat straw	26.50	Brewers' grains	24–30
Barley straw	24.47	Corn cobs	31.34
Oat straw	24.84	Linseed meals	13.49
Pea straw	17.11	Gluten feed	17.16
Corn stover	15–22	Gluten meal	4.50
Clover hays	11–15	Dried distillery feed (atlas gluten feed)	13
Corn meal	6.02	Rape cake	5–08
Whole oats	15.55	Sugar beets	9–12

Table VI.—*Galactan.*

[Determined by E. B. Holland.]

Cattle feeds.	Galactose.	Galactan.	Cattle feeds.	Galactose.	Galactan.
COARSE FODDER.	*Per cent.*	*Per cent.*	CONCENTRATED FEEDS—con'd.	*Per cent.*	*Per cent.*
English hay	1.01	0.91	Green soja bean	0.67	0.60
High grown salt hay	.93	.84	Black soja bean	.92	.83
Black grass	.71	.64	Bush lima bean	.79	.71
Corn stover	.76	.68	Pole lima bean	.66	.59
Oat straw	.81	.73	Black wax bean (dwarf)	.52	.47
Rye straw	.63	.57	White pot bean	.53	.48
Fodder millet	.95	.86	Horse bean	1.83	1.65
Canada beauty pea fodder	3.09	2.78	Canada beauty pea	.63	.57
Medium red clover fodder	3.73	3.36	Prussian blue pea	.75	.68
Alsike clover fodder	4.25	3.33	English gray pea	.84	.76
Mammoth clover fodder	3.77	3.39	Little gem pea	1.16	1.64
CONCENTRATED FEEDS.			Wonder pea	1.62	1.46
			Pea meal	2.69	2.42
Corn meal	.05	.05	Vetch seed	1.17	1.05
Wheat meal	.23	.21	Serradilla seed	.66	.59
Oat meal	.81	.73	Medium red clover seed	2.77	2.49
Barley meal	.55	.50	Mammoth clover seed	3.63	3.27
Wheat bran	.43	.39	Crimson clover seed	3.49	3.14
Millet meal	.67	.60	Alsike clover seed	6.96	8 05
Linseed meal	1.31	1.15	Sweet clover seed	6.00	5 40
Cotton-seed meal	.63	.57	White clover seed	10.08	9.07
Rice meal	1.04	.94	Alfalfa seed	5.23	4.71
Rape seed	1.07	.96	White lupine seed	13.84	12 46
Brewers' grain	.56	.50	Blue lupine seed	16.29	14.66
Malt sprouts	.43	.39			
Dwarf horticultural bean	.68	.61			

Many of the substances tested show less than 1 per cent of galactan, and we are not certain in many cases, because of the small amount of precipitate obtained, whether the material weighed really was mucic acid or partially decomposed organic matter. All substances, therefore, containing less than 1 per cent of galactan may be for the present characterized as doubtful. To settle the question of the presence or absence of very small amounts of galactan, we shall either be obliged to still further perfect the method or work with larger quantities.

The results as given above show the presence of very small amounts of galactan in the nonleguminous coarse fodders and seeds. In the leguminous plants from 3 to 4 per cent are present, while in case of the leguminous seeds, several varieties of beans and peas appear to contain very limited quantities, but the larger number of such seeds tested shows from 1½ to as high as 14 per cent. With the exception of the lupines, the clover seeds contain the largest amounts, the seeds of the white variety containing 9 per cent.

The above results are merely a report of progress. They show, however, that the galactans are not so widely distributed nor present in such large quantities as are the pentosans, and therefore do not play such an important part as do the latter in the process of nutrition.

The data in the following table show the relations existing between the known and unknown nitrogen-free matters in some common substances:

Difference between estimated carbohydrates and extract matter.

Substances.	Corn meal.	Middlings.	Lupine.
	Per cent.	*Per cent.*	*Per cent.*
Starch (diastase method)	64.94	20.40	2.23
Pentosans (phloroglucin method)	5.67	19.50	9.54
Galactan	None.	None.	14.87
Total	70.61	39.90	26.64
Extract matter	83.35	58.72	40.38
Soluble carbohydrates and unaccounted for	11.74	18.82	13.74
Allowing for soluble carbohydrates	3.00	3.00	3.00
Still unaccounted for	8.74	15.82	10.74
Percentage of total extract unaccounted for	10.60	26.90	26.60

Stone[1] has already called attention to this fact. Of the cause of this difference we are at present ignorant. A portion of it can possibly be accounted for because of imperfect methods for the estimation of carbohydrates. In materials of a woody nature, containing large amounts of crude fiber, it is known that the action of dilute acid and alkali removes a considerable portion of the lignin acids so that they are reckoned with the extract matter. It seems, however, quite probable that other carbohydrates or similar substances exist, which have not as yet been identified.

Estimation of Pentosans.

Councler[2] has suggested that, instead of phenylhydrazin, phloroglucin be employed for the precipitation and estimation of furfurol obtained by the distillation of various substances, with dilute hydrochloric acid. Kruger and Tollens[3] have further studied and perfected the method, and recommended it as reliable for the estimation of pentosans in various coarse fodders, grains, and vegetables.

The phloroglucin, like the phenylhydrazin method, is based on the fact that the pentosans (araban, xylan, etc.) differ from other carbohydrates in that they yield furfurol instead of levulinic acid, upon digestion with moderately dilute hydrochloric or sulphuric acid. The first step necessary in both processes, for a quantita-

[1] Journal of Am. Chemical Society, Vol. XIX, 4.
[2] Chemikerztg., 1894, No. 51.
[3] Zeitsch. für ang. Chem., 1896, Heft II.

tive estimation, is the conversion of the pentosans into furfurol and its separation from the resulting by-products.

On account of its greater simplicity, the phloroglucin method is to be preferred. We considered it advisable, therefore, still further to compare the two methods with substances containing different quantities of pentosans.

PHLOROGLUCIN METHOD DESCRIBED.

Three grams of the material are brought into a 10-ounce flask, together with 100 cc of 12 per cent hydrochloric acid (sp. gr., 1.06), and several pieces of recently heated pumice stone. The flask, placed upon wire gauze, is connected with a Liebig condenser, and heat applied, rather gently at first, using a gauze top to distribute the flame, and so regulated as to distill over 30 cc in about ten minutes from the time that boiling begins. The 30 cc driven over are replaced by a like quantity of the dilute acid, by means of a separatory funnel, and the process so continued so long as the distillate gives a pronounced reaction with anilin acetate on filter paper (a few drops of anilin in a little 50 per cent acetic acid). To the completed distillate is gradually added a quantity of phloroglucin[1] dissolved in 12 per cent hydrochloric acid, and the resulting mixture thoroughly stirred. The solution first turns yellow, then green; and very soon an amorphous greenish precipitate appears, which grows rapidly darker till it finally becomes almost black. The solution is made up to 500 cc with 12 per cent hydrochloric acid, and allowed to stand over night. In case there is very little furfurol in the substance tested, and the resulting distillate consequently small, it is best to add sufficient 12 per cent hydrochloric acid to the distillate before adding the phloroglucin solution, so that, upon the addition of the latter solution, the resulting mixture will contain approximately 500 cc.

The amorphous black precipitate is filtered into a tared Gooch crucible through an asbestus felt, washed with 100 cc of water,[2] dried to constant weight by heating three to four hours at 100° C., cooled and weighed, the increase in weight being reckoned as phloroglucid. To calculate the furfurol from the phloroglucid,[3] use the following table:

Calculation of furfurol.

Total weight of phloroglucid obtained.	Divided by, equals furfurol.	Total weight of phloroglucid obtained.	Divided by, equals furfurol.
0.20 gram	1.820	0.34 gram	1.911
0.22 gram	1.839	0.36 gram	1.916
0.24 gram	1.856	0.38 gram	1.919
0.26 gram	1.871	0.40 gram	1.920
0.28 gram	1.884	0.45 gram	1.927
0.30 gram	1.895	0.50 + gram	1.930
0.32 gram	1.904		

Furfurol ÷ by grams substance taken × 1.84 = pentosans.
Furfurol ÷ by grams substance taken × 1.65 = xylan.
Furfurol ÷ by grams substance taken × 2.03 = araban.

[1] Dissolve twice as much dry phloroglucin as furfurol expected in about 50 cc of 12 per cent hydrochloric acid. Bring the hydrochloric acid into a water bath, and stir thoroughly till the phloroglucin goes into solution.

[2] By allowing the suction pump to work for some time after the filtration is completed, the drying is greatly facilitated.

[3] The phloroglucid is a complex substance, of uncertain formula. It contains 63 to 64 per cent of carbon and from 3.6 to 4.2 per cent of hydrogen. The factors for calculating the amount of furfurol from the phloroglucid were obtained after experimenting with known amounts of pure furfurol and phloroglucin.

PHENYLHYDRAZIN METHOD.

Proceed exactly as in the phloroglucin method until all of the furfurol is distilled over. Pour the combined distillates into a 700 to 800 cc beaker. If the distillates be less than 400 cc, add 10.2 grams of sodium chlorid for each 50 cc less than that volume, and complete the volume with water to 400 cc. Neutralize exactly with sodium carbonate, covering the beaker with a watch glass during the process, and make up to 500 cc; add 10 cc of phenylhydrazin[1] solution, precipitating the furfurol as furfurol hydrazone.[2] Stir thirty minutes, preferably with an automatic stirrer, and filter the solution, using the necessary suction, into a so-called sugar tube about three-quarters inch in diameter and 6 to 7 inches long, drawn out at the lower end and filled with glass wool. Remove the adhering particles with a feather, and do not use over 100 cc of wash water. The drying can be very much hastened by allowing the filter pump to run for some time after the washing has been completed.

Complete the drying in a specially constructed air bath[3] at 55° C. To accomplish this, a partial vacuum is made by drawing air through the tube by aid of a suction pump, the air supply being regulated by pinchcocks. The air before entering the tube is conducted through sulphuric acid to dry it, and through vertical glass tubes containing small pieces of marble, to remove any sulphuric acid that might be carried over mechanically. Cool the tubes in a desiccator and weigh. Dissolve out the precipitate with hot alcohol and reweigh, and consider the loss in weight as furfurol hydrazone.

FACTORS.

Furfurol hydrazone × .516 + .0104 ÷ weight of substance taken = furfurol.
Furfurol × 1.84 = pentosans.
Furfurol × 1.65 = xylan.
Furfurol × 2.03 = araban.

TABLE VII.—*Results of comparative tests.*

Analysts.	1. Corn meal.		2. Lupine seed.		3. Wheat middlings.	
	Phenylhydrazin method.	Phloroglucin method.	Phenylhydrazin method.	Phloroglucin method.	Phenylhydrazin method.	Phloroglucin method.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
W. H. Krug, U. S. Dept. of Agrl	4.78	.....	[1]12.23	.....	18.19	.....
C. D. Howard, N. H. Exp. Stat	.....	5.40	.....	8.89	.....	18.50
	(5.94)	.....	(9.91)	.....	(19.63)	.....
C. A. Brown, Jr., Penn. Exp. Stat	5.11	5.60	9.04	9.80	18.77	19.30
E. B. Holland, Mass. Exp. Stat	4.72	6.02	9.45	9.94	20.23	20.51
Average	4.87	5.67	9.24	9.54	19.06	19.50

[1] Excluded from average.

In corn meal the phloroglucin method gives noticeably higher results. Mr. Wiley reports a similar experience with wheat, securing 5 per cent of pentosans by the phenylhydrazin and 8 per cent by the phloroglucin method. A sample of wheat tested in our laboratory gave 6.90 per cent by the former and 8.78 per cent by the latter method. We are at present unable to account for this difference. Excepting the grains, it can be said that the phloroglucin gives only very slightly higher results than the phenylhydrazin method. Mr. Brown, in Mr. Frear's laboratory, had

[1] Twelve grams of recently distilled phenylhydrazin and 7.5 grams of glacial acetic acid filled to 100 cc with water and well shaken.

[2] After the addition of the phenylhydrazin the solution should react only *very slightly* acid. This is very important. If necessary add the required acetic acid.

[3] See Twelfth Report Massachusetts Experiment Station, 1894, p. 188.

difficulty in securing a clear filtrate after stirring the phenylhydrazin precipitate for a half hour and filtering. By allowing the filtrate to stand forty-eight hours and then filtering again he secured, after deducting a blank for the decomposition of the phenylhydrazin itself, 0.039 gram, which he assumes to be phenylhydrazin. In the figures inclosed in the above table, he has added this to his original weight, and in making the calculations for pentosans omitted to add 0.0104, as the formula prescribes. It is to be confessed that by this means his results are brought nearer those obtained by the other method, yet because of the way in which the method was made up this would hardly be allowed.

RECOMMENDATION.

We recommend the phloroglucin method, as described above, as the provisional method for the estimation of furfurol in agricultural products.

SUGGESTION.

It is suggested that further study be given to these methods, in order to ascertain their apparent lack of agreement when employed for the estimation of pentosans in the grains.

ESTIMATION OF GALACTAN.

For the estimation of galactan the following method is recommended:

Bring 3 grams of the substance into a beaker about 5.5 cm in diameter and 7 cm deep, together with 60 cc of nitric acid of 1.15 specific gravity, and evaporate the solution to exactly one-third of its volume in a water bath at a temperature of 94° to 96° C. After standing twenty-four hours, add 10 cc of water to the precipitate and allow it to stand another twenty-four hours. The mucic acid has in the meantime crystallized, but is mixed with considerable material only partially oxidized by the nitric acid. The solution is therefore filtered, washed with 30 cc of water to remove as much of the nitric acid as possible, and the filter and contents brought back into the beaker. Thirty cc of ammonium carbonate solution, consisting of 1 part ammonium carbonate, 19 parts water, and 1 part strong ammonia are added, and the beaker brought into a water bath and heated gently for fifteen minutes. The ammonium carbonate takes up the mucic acid, forming the soluble mucate of ammonia. The solution is filtered into a platinum or porcelain dish, and the residue thoroughly washed with water to remove all of the mucate of ammonia. The filtrate is evaporated to dryness over a water bath, and 5 cc of nitric acid of 1.15 specific gravity are added, thoroughly stirred and allowed to stand for thirty minutes. The nitric acid decomposes the ammonium mucate, precipitating the mucic acid, which is collected on a tared filter or Gooch crucible, washed with from 10 to 15 cc of water, then with 60 cc of alcohol, and quite a number of times with ether, dried at 100° for a short time, and weighed. The mucic acid multiplied by 1.33 gives galactose, and this multiplied by 0.9 gives galactan.

Papers relating to the analyses of feeding stuffs were called for and the following report was made.

PRELIMINARY REPORT ON THE ESTIMATION OF FAT IN FODDERS AND GREEN MATERIALS.

By B. W. KILGORE.

"Ether extract" is known by all chemists not to represent the true fat in fodders, hays, and green feeding materials. That it would be highly desirable and important to be able to determine pure fat instead of ether extract in this class of substances is also recognized by all who have to do with them in a way where the question of a knowledge of their composition is involved. So far as we know, no method for obtaining pure fat from these materials has yet been worked out. With-

out going into a consideration of the character of the substances in ether extract other than fat or of their possible value in nutrition; without considering the effect of a method giving pure fat on the large accumulation of data in the past in the analysis of foods and feeding stuffs and in feeding experiments for beef and butter, or what would be the effect of such a method on this class of work in the future; and without mentioning the numerous failures which have been encountered in the course of a study of this subject during the past eighteen months (since March, 1896), or giving the reasons which led to the procedure in the method, I merely propose to make a preliminary statement to the association of the success I have had in working out a method for the estimation of pure fat in the class of materials referred to. Work on this subject is still in progress in our laboratory, and we ask that the matter be left to us until we have had time to perfect the method and obtain sufficient data for a detailed report.

There are two methods of procedure, both giving about the same results, which appear to be quantitative. Most of our work has been done on the soja-bean plant, which gives quite an impure extract, but enough other materials has been experimented upon to make us believe that the methods will be good for all substances.

The first procedure, which gives not the fat but the fatty acids, is briefly as follows:

Extract the material thoroughly with ether or benzine, and afterwards with alcohol if necessary. (Especially with old materials and leguminous plants containing lecithin, alcohol will remove fat which is not soluble in ether or benzine.) Remove the solvent, saponify with alcoholic potash or soda; evaporate the alcohol, take up the soap with hot water, decompose with hydrochloric acid, add some woolly asbestus, and stir to collect the fat; filter and wash with warm water, partially dry on water bath, and extract with carefully fractioned benzine of 30°–50° C. This gives apparently quite pure fatty acids, usually of a slightly yellowish color.

The second procedure is to extract material with benzine through proper amounts of anhydrous copper sulphate, neutral precipitated calcium carbonate, calcium chlorid, or plaster of Paris. These substances hold back the chlorophyll and coloring matter and yield very nice extracts. Some other salts act in the same way as the above, but possess no advantage over them. These salts do not render the coloring matter of plants insoluble in ether.

DISCUSSION OF ESTIMATION OF STARCH.

Mr. WILEY. I have been as much interested in this paper as in anything else that has been presented, not only because I take very particular interest in the question, but because it is the beginning of the themes which are to be the great discussions of this association in the future. We have practically exhausted the subjects of potash, nitrogen, and phosphoric acid. In subsequent years we may make a few amendments in processes, but it is perfectly certain that unless some new discoveries be made in analytical chemistry our methods in these respects are practically fixed, and the future work of the association lies in those lines which have been neglected in the past. This question of food products, is one of the greatest. The discussion of our food materials in their economic uses, as well as their actual composition, is one of the great problems now confronting the agricultural chemists of the world.

You will be struck by this fact, that the method which is recommended by the referee as the one which should be followed as a provisional method is the one which gives almost the widest variations in

results in the hands of the analyst. Now, I appreciate the reasons which led the referee to make this recommendation, and I do not think I could have done otherwise than make the same one myself; but at the same time we must admit that, even in the hands of the experienced analyst, the widest results are obtained in the application of this method. Now, the diastase method rests upon the assumption that this ferment will bring into solution all the starch particles. This is a fundamental error to begin with. It may or it may not; usually it does not. In the first place the action of diastase is conditioned by a great many things in the environment—the temperature, the degree of fineness (which is very important), and the character of the diastase itself. We can not all use the same solutions. We have to make them as we use them, and there are differences in malt. The result is that half a dozen men of this association, in buying malt or preparing it themselves, get solutions which are distinctly different in their hydrolytic properties. Here is a fundamental error of the diastase method—that we do not all work with the same materials or reagents. We prescribe our diastase solution to be made according to a certain method, and yet every man who makes it may have a solution of a different strength, although he follows exactly the directions of the referee.

Another thing, there is no way of knowing that the starch is dissolved except by actual examination. I do not believe there is a member here who can take a ground cereal and extract the starch from it by a single treatment with diastase. The only safe way is to take a portion, stain it, and examine with the microscope. If you get a blue color, repeat the treatment with diastase.

Now, with regard to the operation of diastase upon pentosans, I am glad to have the matter brought before the association, because Mr. Krug and I have paid a good deal of attention to it. Mr. Krug has found, as other investigators of the matter have, that the action of diastase upon pentosans is not a very vigorous one, and, as the referee very aptly suggested, it may be due to the water rather than to the diastase. But, at least, of all reagents used for separating starch from its compounds in cereals, diastase acts least vigorously on the pentosan and other insoluble carbohydrate bodies not starch, present. For that reason, it is to be recommended above all other reagents which have been proposed for this purpose. A paper was published not long ago by König, in Germany, claiming that diastase does dissolve large percentages of pentosans, and this theory we subjected to a very careful and critical examination. Mr. Krug found this claim was a mistake; that while diastase did act to a certain extent in dissolving the pentosan bodies present it was to a very limited extent. On the other hand, we found that salicylic acid under certain conditions would dissolve as high as 90 per cent of the pentosans present. So we have modified our method with regard to starch determinations in this way, that inas-

much as all reagents which dissolve starch do dissolve pentosans to a certain extent it is useless to seek for a reagent which will not.

Then the question arises: Should we not pursue a method which will get the starch at once into solution without respect to the action of the reagent on the pentosan bodies? Our modification, therefore, has been to eliminate the slowly acting bodies and take strongly acting bodies like salicylic acid under high pressure, and then, after determining the total copper which is precipitated in this solution, determine the pentosan bodies therein, or the galactan bodies if they have been acted upon, and deduct from the total the sugar equivalent to these bodies.

The question, of course, of the amount of other bodies yielding dextrose from the fiber which may be present has yet to be considered. Nevertheless, we use practically the diastase method in our determinations. We find, as the referee finds, that we can not conscientiously make any other recommendation at the present time, but we simply insist upon the fact that all the starch bodies shall be brought into solution.

Another question is important. The estimation of potash, of phosphoric acid, and of nitrogen is easy, but it is a difficult thing to estimate starch. In the first place, ordinarily the quantity of material acted upon is small and then only a very small aliquot of that material is used, so that we get in the end only 150 to 200 milligrams as representing the total weight of the material. Hence, any errors in manipulation are greatly magnified before the result is reached, and there is no manipulation which the agricultural chemist is called upon to engage in which requires so much skill, delicacy, and care in every detail as the estimation of starch.

Then we have wide differences in the methods of determining copper. One chemist determines it by the volumetric method; another will precipitate and weigh the copper as oxid after ignition; another will, after precipitation of the copper, bring it into solution and precipitate it electrolytically and weigh it as metallic copper. Theoretically, all these methods should yield good results, but it seems to me that in practice we ought to use a uniform method. We have long since abandoned in our laboratory any attempt to estimate copper volumetrically. In the sugar factory, the volumetric method is to be recommended, because it is quick and reasonably accurate for control purposes, but for all such purposes as ours for fixing methods the volumetric method must be absolutely eliminated; that we must accept as true.

With regard to the differences between estimations as oxid of copper and metallic copper, I think experience leads us to prefer in every respect metallic copper, and I believe the association should adopt that as its method. The copper should be deposited electrolytically and weighed as metallic copper. If a great many of these determinations be made it requires very little more time than the other methods I have mentioned. The analyst working through the day and preparing,

say, twenty of these solutions, can connect them over night with the current and in the morning will find the copper precipitated. Therefore we strongly recommend this electrolytic method for the determination of copper.

The bodies which yield furfurol on distillation are far from being simple in their composition. Here is another great trouble we experience. We assume that all the furfurol yielded is due to a definite body, araban or xylan. Now that is a great error, I am sure. If all kinds of fibers be treated for pentosan there will be found a certain quantity of pentosan bodies which are dissolved very rapidly by alkaline solution from these bodies. There is another large quantity of substance, the furfurologenes, which is not dissolved at all, or only slowly, by alkaline bodies. Krug has shown that fibers treated for several hours at high pressure with caustic soda of a certain strength contain, still undissolved, large quantities of bodies yielding furfurol. So we have here very great differences in these bodies. Bevan and Cross have called attention to this and have also proposed the name furfuroid bodies for those which are different from araban, xylan, etc. These are probably substances which have different degrees of solubility. Now what is the relation of these bodies to the original amount of furfurol yielded?

We have found this, that there is no unaccounted-for matter, or at least only a trace, in cereals if all the starch and all the furfurol-yielding bodies calculated as xylose be determined. That practically completes the whole of the cereal material. We have in cereals little galactan. We have a certain amount of soluble sugars which pass into solution, mineral salts, organic compounds, phosphorus, sulphur, etc., and a careful estimation of the starch of a cereal by the diastase method, together with a determination of the other ingredients by approved methods, yields practically 100 per cent. Mr. Krug has obtained results, working with the greatest care, on large numbers of cereals, which come almost as near footing up 100 as ordinary mineral analyses. When it comes to the other feeding stuffs and fibers, the presence of this large amount of a furfurol-yielding body not soluble in alkali under high pressure complicates the determination; but I have no doubt when this relation is established, i. e., the furfurol relation to this other class of bodies, this gap will be filled up.

There is one recommendation of the referee which I hope the committee will take into serious consideration, and that is in regard to shortening the time of conversion. It is highly important that the dextrinoid bodies separated in starch determinations be converted into dextrose, because dextrose is the body we determine. Now, while two hours' boiling will be sufficient in most cases, I do not think the additional hour is going to do any harm. If the time be diminished, the liability to error is increased. Of course there is a difference between boiling and heating to the boiling point. At any rate, the conversion must be complete before the dextrose can be determined.

I do not know, Mr. President, of any more important subject than the one which the referee has so admirably brought before us. The statements in his paper demand and should receive the serious consideration of every member of our association. I should like to see more of our members giving their attention to the investigation of these important problems. There is plenty of room for investigation; nobody has preempted the whole field.

Mr. LINDSEY. It has been very gratifying to me to have Mr. Wiley bring up this matter. I realize that my paper is imperfect, since we have not had time to go over all the points we desire to treat. I want to make one explanation, however, and that is, he has suggested in his method, which gives not only starch but also galactans and pentosans, that all these be taken from the total, and from that be taken the amount of pentosans and galactans, and the remainder be called starch. That I can not accept, because I am convinced that by this pressure a portion of the galactan and pentosans is destroyed, so that we can not get them into bodies which will reduce copper. I realize the difficulty of bringing starch into solution by the aid of diastase, yet I am convinced that at present we can get safer results by the diastase method than by any other, when we are attempting to estimate pure starch; and if Mr. Wiley will refer to the table he will find that Mr. Krug (in Table 6, I think) got very similar results with the diastase method and the Märcker method. I can not see how that is possible. You will also notice that in wheat middlings he got 31 per cent with the diastase method and 35 per cent with the Märcker method. I think his diastase must have worked pretty severely in that case. I can not reconcile his figures at all. I believe the diastase method, at the present time, is the safest method.

In describing the diastase method I have recommended that the substance be powdered. I am convinced that in passing it through a millimeter sieve we do not get it as fine as we ought. To get all the starch acted upon by the diastase, the substance should be put into the form of a powder. I do not doubt that the Department of Agriculture got really more starch than we did. But by the ordinary method, as it is recommended by Märcker and others, the statement is: Treat with diastase solution until the substance no longer gives iodin reaction for starch. That we followed out. I believe by grinding it fine we can get more starch. With reference to the methods for precipitation of copper, I think Mr. Wiley is right.

Mr. WILEY. A cereal can be treated by the diastase method until by the ordinary test it gives no starch reaction at all, yet if examined by the microscope it is found that plenty of starch is left. It is because these starch granules are entirely surrounded by proteid and other incrusting matters that they escape solution. This fact led Mr. Krug and myself to try an interesting modification which we think will be of great benefit, and that is to digest previously with pepsin before using the diastase at all. Our attention was called to pepsin by a French

chemist, Mr. Lindet, who proposes to estimate starch directly as starch, and, by the way, his method is an excellent one for technical purposes. He digests his cereal with pepsin and then washes on silk cloth, collects the starch, dries and weighs it; and for technical purposes that is the best method. Mr. Krug has tried this method very thoroughly and finds the amount of fiber held by the starch is not very different from the amount of starch which remains in the fiber. It is an excellent technical method and a fairly good analytical one. This suggested to us the previous solution of this proteid matter before applying the diastase.

Mr. LINDSEY. Would it not be wise to digest all the material, everything, with pepsin?

Mr. WILEY. We don't know but it would. We have not gone far enough to say, but our results are very promising.

Mr. Bartlett asked if longer boiling in water would not be effective in getting at the starch.

Mr. WILEY. I think no amount of boiling would separate the starch from this proteid film. While boiling is an excellent thing for most starch granules, it can not reach the trouble we have here. No amount of boiling will disintegrate these granules.

Mr. WINTON. Where only a few determinations are to be made a very good substitute for diastase is ordinary human saliva. This method was suggested by Professor Chittenden, of Yale, who has worked out very carefully the diastatic action of saliva and has given exact instructions for securing its maximum action. I tried this method eight or ten years ago and found the starch was very completely brought into solution. The action was, if anything, quicker than that of diastase. We have no correction for sugar to make in this reagent.

Mr. WILEY. The trouble with saliva is that it is rather difficult to filter. It takes a long time to get it through. Of course, this use of saliva is an old one; we have tried it also, but did not think its activity, except in perfectly free granules, was sufficient. Then we had great difficulty in filtration; even diastase does not filter any better than it should.

Mr. Van Slyke moved to postpone discussion of the next three papers relating to foods until all three had been read. Adopted.

Mr. Kilgore presented informally a preliminary report on some work he had been doing in the estimation of fat. The chair suggested that Mr. Kilgore write up this statement in form for publication in the proceedings, which Mr. Kilgore agreed to do.[1]

Mr. Hills exhibited some blue prints of a mill which he had in use for grinding fodder samples, and described its operation.

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[1] The paper by Mr. Kilgore is found on page 98.

The chair called for the report on food legislation. This was read by Mr. Wiley, as follows:

REPORT ON FOOD LEGISLATION.

To the President and Members of the Association of Official Agricultural Chemists.

GENTLEMEN: Your committee, appointed to consider the subject of pure-food legislation, begs to make the following report:

The importance of a general pure-food law for the District of Columbia and the Territories of the United States, and for foods passing from one State to another, is admitted by all who have devoted any attention to the subject of pure-food legislation. The authority of the National Congress can not go beyond the limits mentioned above. Already the National Legislature has passed a local food law for the District of Columbia, which in many of its provisions would be suitable for general legislation. It is important, however, that a national food law which will apply to the whole country within the limits mentioned above should be typical in character and become a basis upon which the State legislatures might model the local laws for the several States. Not only is it evident that legislation in regard to pure foods, both national and State, is of the utmost importance, but it is also at once evident that there should be a general agreement in the provisions of the laws enacted by the General Government and by the States. Your committee would recommend that this association urge upon Congress the passage of a pure-food law, the essential provisions of which are the same as the Paddock law, which passed the Senate on March 9, 1892, but failed to come to a vote in the House of Representatives. Your committee would recommend a reintroduction of the bill with a few slight modifications which follow in the appended copy.[1] Your committee is of the opinion that a general bill regulating food adulteration would be far more effective than the passage of particular acts, such as those relating to the manufacture and sale of oleomargarine, filled cheese, etc. The whole subject of food adulteration should, in the opinion of your committee, be treated in a broad and scientific spirit by the National Legislature in such a way as to secure the enactment of a law which, in its scope and method of application, could serve as a model for similar laws in all the States.

In conclusion, your committee would recommend that if favorable action be taken upon the report herewith submitted, the same be transmitted to the proper committees of the national House of Representatives and of the Senate, with the indorsement of this association favorable to a speedy action along the lines mentioned.

Respectfully,

H. W. WILEY.
H. A. HUSTON.
A. S. MITCHELL.

The referee on dairy products read the following report:

REPORT ON METHODS FOR THE ANALYSIS OF DAIRY PRODUCTS.

BY L. L. VAN SLYKE, REFEREE.

The cooperative analytical work on dairy products has been limited this year to a study of methods used for determining the casein and albumin in cow's milk.

Some special work has been done by the referee also in order to call attention to some deficiencies existing in the present statement of our official method for determining milk solids.

[1] See pure-food bill, introduced into the House of Representatives by the Hon. Mr. Brosius. H. R. 5441.

The referee was authorized by vote of the association to study methods for identifying filled cheese and milk adulterants used in the form of milk preservatives. At the suggestion of the chairman of the committee, appointed to report methods for the detection of food adulterants, I have made the present report cover dairy products in general.

Determination of Casein and Albumin in Cow's Milk.

There is probably no class of compounds in agricultural chemistry about which there is more variation of statement than the nitrogen compounds of cow's milk. Thus, Duclaux declares there is only one, while Halliburton says unqualifiedly, "The proteids which occur in milk are two in number." Wiley names four, but states that the number and kind of proteid bodies are not known with definiteness. Sebelien says that milk contains globulin, and Halliburton says this is a mistake. New nitrogen compounds are frequently reported as being found in milk.

The referee did not have it in mind to harmonize these disagreements or settle these disputed questions, but simply to assume as true what is most palpable, and study methods based on these assumptions.

From general chemical behavior we can assume, with our present light, that cow's milk contains at least three kinds of nitrogen compounds. First in quantity is casein, which is coagulated by acids, rennet, rennet-like ferments, by saturation with neutral salts like magnesium sulphate, sodium chlorid, etc., or through precipitation by various other metallic salts. Casein can not be separated from other milk constituents by heat alone. The nitrogen compounds remaining in milk after the removal of casein appear to be of two kinds, one kind called albumin and rendered insoluble by heating at 75° C. or higher. Then there always remains, so far as my own experience goes, a class of nitrogen compounds which is not affected either by heat or by the reagents used to remove casein.

To separate and estimate the casein and albumin of milk, two methods have been used for study: First, the provisional method adopted last year, that is, precipitation of casein by acetic acid and determination of albumin in filtrate by boiling; and second, precipitation of casein by saturation with magnesium sulphate and determination of albumin in acidified filtrate. The latter method has been carried out as follows: To about 5 grams of milk, 50 cc of solution of magnesium sulphate, saturated at 40° to 45° C., are added and the mixture heated to 40° to 45° C. until the precipitate completely separates, leaving the surrounding liquid clear. The precipitate is filtered, washed two or three times with warm saturated solution of magnesium sulphate, and then treated by the Kjeldahl method for the determination of nitrogen. To the filtrate is added 0.3 cc of a 10 per cent solution of acetic acid and the temperature is raised to boiling until the albumin completely precipitates. The precipitate is filtered, washed with saturated magnesium sulphate solution, and then treated by the Kjeldahl method for the determination of its nitrogen. Some work has been done also by the Geneva Station on a modification of the provisional method for determining albumin which will be described later.

Three different samples of milk were sent on different dates, as follows:

On June 14, to the Indiana, Iowa, and Cornell University experiment stations.

On July 26, to the Department of Agriculture at Washington, and to the Wisconsin Experiment Station.

On September 13, to the Cornell University, Maine, Delaware, Kentucky, Vermont, and Ottawa (Canada) experiment stations, and to the Department of Agriculture.

These samples were examined at the Geneva Experiment Station in every case. The work was performed by the following chemists: Cornell University, C. G. Cavanaugh; Department of Agriculture, G. E. Patrick and T. C. Trescot; Geneva, J. A. Le Clerc and L. L. Van Slyke; Indiana, H. A. Huston; Iowa, I. J. Mead; Kentucky, M. A. Scovell and A. M. Peter; Maine, J. M. Bartlett; Ottawa (Canada), F. T. Shutt; Vermont, C. H. Jones; Wisconsin, Alfred Vivian.

The samples sent out consisted of freshly separated skim milk, and contained for each liter 1 cc of 40 per cent formaldehyde solution to keep the casein from undergoing change. In this connection, it was desired to ascertain whether the presence of formaldehyde in this amount would affect the determination of casein or albumin by either method, and also whether on long standing the casein would undergo change in the presence of formaldehyde.

The following results indicate that the presence of formaldehyde in the comparatively large proportion used has no influence upon the precipitation of the casein or albumin by the methods employed.

Effect of formalin in milk.

Quality of milk.	Percentage of casein by provisional method.	Percentage of casein by magnesium sulphate precipitation.	Percentage of albumin by provisional method.	Percentage of albumin by magnesium sulphate method.	Percentage of albumin by modified provisional method.
In milk containing no formalin	(a) 2.96	(a) 3.24	(a) 0.388	(a) 0.469	(a) 0.463
	(b) 2.98	(b) 3.26	(b) .400	(b) .475	(b) .475
	(c) 3.01	(c) Lost.	(c) .425	(c) .500	(c) .481
	(d) 3.04				
	(e) 3.05				
	(f) 3.06				
Average	3.02	3.25	.404	.481	.473
In milk containing formalin	(a) 3.01	(a) 3.21	(a) .406	(a) .413	(a) .425
	(b) 3.01	(b) 3.22	(b) .406	(b) .444	(b) .444
	(c) 3.04	(c) 3.24	(c) .413	(c) .450	(c) .463
	(d) 3.05				
	(e) 3.06				
	(f) Lost.				
Average	3.03	3.22	.408	.436	.444

That formalin is a most excellent preservative for keeping samples of milk for analysis our results, presented below, show conclusively. The sample of milk, which was sent out on June 14, was analyzed for casein and albumin at six different times between June 14 and July 26. The sample during these several weeks stood in the laboratory, where it was fairly warm.

The amount of casein as found by the provisional method was as follows:

(1) Sample sent out June 14:

	Per cent.
June 14	2.54
17	2.57
19	2.62
25	2.59
July 19	2.59
26	2.66

(2) Sample sent out July 26:

	Per cent.
July 26	2.62
Aug. 2	2.68
12	2.61
25	2.59

The results obtained by the magnesium sulphate method were equally uniform. The albumin determinations were less uniform, but fairly good. We may safely conclude that formaldehyde will keep milk in perfect condition for analysis for a reasonable length of time, and especially if any precautions whatever be taken to keep the milk in a fairly cool place.

Therefore, any difference existing in the results obtained by different workers can not be due to the fact that the milk underwent change before being examined; and, moreover, the work was completed in every case with reasonable promptness after the receipt of the samples.

MODIFICATION OF PROVISIONAL METHOD FOR DETERMINATION OF ALBUMIN.

Some time ago we noticed irregularities in determining albumin by the provisional method which seemed to point to low results. It occurred to us that under some conditions the amount of acetic acid used for precipitating casein might be sufficient to hold some albumin in solution. We therefore made a series of comparisons, neutralizing by caustic alkali the filtrate from casein precipitation and then adding 0.3 cc of 10 per cent solution of acetic acid. The latter method quite uniformly gave higher results, varying somewhat in different milks. The data for the comparisons are here presented.

Results from sample No. 1.

Stations.	Percentage of casein by provisional method.	Percentage of casein by magnesium sulphate method.	Percentage of albumin by provisional method.	Percentage of albumin by magnesium sulphate method.
New York (Geneva)	(a) 2.44	(a) 2.65	(a) 0.225	(a) 0.275
	(b) 2.54	(b) 2.66	(b) .238	(b) .300
	(c) 2.54	(c) 2.70	(c) .244	(c) .325
	(d) 2.54	(d) 2.71	(d) .256	(d) .325
	(e) 2.62	(e) 2.76	(e) .262	(e) .344
Average	2.54	2.70	.245	.314
Indiana	(a) 2.62	(a) 2.93	(a) Lost.	(a) .371
	(b) 2.63	(b) 3.02	(b) .401	(b) .358
	(c) 2.63	----------	(c) .379	----------
Average	2.63	2.97	.390	.365
Iowa	2.66	2.78	.219	.197

Results from sample No. 2.

Stations.	Percentage of casein by provisional method.	Percentage of casein by magnesium sulphate method.	Percentage of albumin by provisional method.	Percentage of albumin by magnesium sulphate method.
New York (Geneva)	(a) 2.59	(a) 2.72	(a) 0.231	(a) 0.281
	(b) 2.60	(b) 2.77	(b) .262	(b) .312
	(c) 2.62	(c) 2.78	(c) .287	(c) .363
	(d) 2.67	(d) 2.79	(d) .306	(d) .400
	(e) 2.68	(e) 2.84	(e) .331	(e) .406
Average	2.63	2.78	.283	.352
Department of Agriculture	(a) 2.56	(a) 2.69	(a) .325	(a) .244
	(b) 2.56	(b) 2.69	(b) .338	(b) .244
	(c) 2.63	(c) Lost.	(c) .344	(c) .244
Average	2.58	2.69	.335	.244
Wisconsin	(a) 2.50	(a) 2.62	(a) .313	(a) .100
	(b) 2.50	(b) 2.63	(b) .314	(b) .120
	(c) 2.54	(c) 2.64	(c) .318	(c) .120
	(d) 2.56	(d) 2.65	(d) .330	(d) .125
Average	2.53	2.63	.319	.116

Results from sample No. 3.

Stations.	Percentage of casein by provisional method.	Percentage of casein by magnesium sulphate method.	Percentage of albumin by provisional method.	Percentage of albumin by magnesium sulphate method.	Percentage of albumin by modified provisional method.
New York (Geneva)	(a) 2.71	(a) 3.04	(a) 0.269	(a) 0.288	(a) 0.362
	(b) 2.76	(b) 3.10	(b) .300	(b) .325	(b) .369
	(c) 2.79	(c) Lost.	(c) .344	(c) .369	(c) .387
Average	2.75	3.07	.304	.327	.373
New York (Cornell)	(a) 3.10	(a) 3.19	(a) .317	(a) .270	.....
	(b) 3.30	(b) 3.23	(b) .360	(b) .317	.....
Average	3.20	3.21	.338	.293	.....
Kentucky	(a) 2.84	(a) 2.85	(a) .32	(a) .34	.....
	(b) 2.86	(b) 2.88	(b) .33	(b) .39	.....
	.....	(c) 2.90	.....	.....	.....
	.....	(d) 2.92	.....	.....	.....
	.....	(e) 2.93	.....	.....	.....
Average	2.85	2.90	.325	.365	.....
Maine	(a) 2.71	(a) 2.91	(a) .275	(a) .425	.....
	(b) 2.71	(b) 2.94	(b) .288	(b) .425	.....
	(c) 2.73	(c) 2.94	(c) .306	(c) .450	.....
Average	2.72	2.93	.288	.431	.....
Vermont	(a) 2.69	(a) 2.69	(a) .252	(a) .42	.....
	(b) 2.72	(b) 2.69	(b) .250	(b) .425	.....
	(c) 2.73	.....	(c) .250	.....	.....
Average	2.71	2.69	.251	.422	.....
Department of Agriculture	(a) 2.63	(a) 2.63	(a) .263	(a) .175	.....
	(b) 2.63	(b) 2.69	(b) .263	(b) .175	.....
	(c) 2.63	(c) 2.75	(c) .275	(c) .175	.....
Average	.....	.....	[1].267	[1].175	.....
	.....	(d) 2.75	(a) .206	(a) .137	.....
	.....	(e) 2.75	(b) .156	(b) .106	.....
	.....	(f) 2.75	(c) .175	(c) .137	.....
Average	2.63	2.72	[2].179	[2].127	.....
Ottawa (Canada)	(a) 2.61	.....	(a) .37	.....	.....
	(b) 2.67	.....	(b) .37	.....	.....
Average	2.64	.....	.37	.....	.....

[1] Washing albumin with cold water.
[2] Washing albumin with hot water.

Summary of analytical results.

Stations.	Percentage of casein by provisional method.	Percentage of casein by magnesium sulphate method.	Percentage of albumin by provisional method.	Percentage of albumin by magnesium sulphate method.	Percentage of albumin by modified provisional method.
Sample 1:					
New York (Geneva)	2.54	2.70	0.245	0.314	.....
Indiana	2.63	2.97	.390	.365	.....
Iowa	2.66	2.78	.219	.197	.....
Average	2.61	2.82	.285	.292	.....
Sample 2:					
New York (Geneva)	2.63	2.78	.283	.352	.....
Department of Agriculture	2.58	2.69	.335	.244	.....
Wisconsin	2.53	2.63	.319	[1].116	.....
Average	2.58	2.70	.312	.298	.....
Sample 3:					
New York (Geneva)	2.75	3.07	.304	.327	0.373
New York (Cornell)	[1]3.20	3.21	.338	.293	.....
Kentucky	2.85	2.90	.325	.365	.....
Maine	2.72	2.93	.288	.431	.....
Vermont	2.71	2.69	.251	.422	.....
Department of Agriculture	2.63	2.72	[2].267 [3].179	[2].175 [3].127	.....
Ottawa (Canada)	2.64	.....	.370	.....	.....
Average	2.72	2.92	.306	.368	.....

[1] Omitted from average.
[2] Washing albumin with cold water; omitted from average.
[3] Washing albumin with hot water; omitted from average.

The data contained in the preceding tables may be summarized as follows:

(1) In the determination of casein we find a good agreement in the duplicates of individual workers by either method.

(2) In the first sample the results of three workers gave casein varying from 2.54 to 2.66 per cent of casein by the provisional method, and from 2.70 to 2.97 per cent by the magnesium sulphate method.

In the second sample the results of three workers gave casein varying from 2.53 to 2.63 per cent by the provisional method and from 2.63 to 2.78 per cent by the magnesium sulphate method.

In the third sample the results of six workers gave casein varying from 2.63 to 3.20 per cent, or, dropping the highest result, from 2.63 to 2.85 per cent by the provisional method and from 2.69 to 3.21 or 3.07 per cent by the magnesium sulphate method.

(3) Comparing the provisional method with the magnesium sulphate method for determining casein, we find that eleven out of twelve workers obtained more casein by the latter method, the difference varying from 0.01 to 0.34 per cent and averaging 0.15 per cent. In only one case was less casein obtained by the magnesium sulphate method and the difference then was only 0.02 per cent.

(4) When we consider that these results are based on the nitrogen determination, and that any error in determining nitrogen is multiplied over six times when we express the results in terms of casein, we may regard the results obtained as very satisfactory, especially when we consider, in addition, that the work was new to most of the analysts.

Every one complained of the tediousness of the magnesium sulphate method, the

time of filtration and washing varying from several hours to three days, as reported by different workers.

While it is evident that acetic acid does not always effect a complete precipitation of casein, yet the difference usually lies below 0.20 per cent, and only in work requiring exceeding accuracy would the tediousness involved in using the magnesium sulphate method justify the added labor. The referee hopes by further work to perfect the provisional method, so that all of the casein may be precipitated.

(5) In the first sample, the results of three workers gave albumin varying from 0.219 to 0.39 per cent by the provisional method and from 0.197 to 0.365 per cent by the magnesium sulphate method.

In the second sample, the results of three workers gave albumin varying from 0.283 to 0.335 per cent by the provisional method and from 0.116 to 0.352 per cent by the other method.

In the third sample, the results of six workers gave albumin varying from 0.251 to 0.338 per cent by the provisional method and from 0.175 to 0.431 by the magnesium sulphate method.

(6) Comparing the provisional method with the magnesium sulphate filtrate method for determining albumin, we find that six workers obtained more albumin by the provisional method and a half dozen other workers obtained more by the other method. However, the average of all results gives albumin by the provisional method as 0.30 and by the other method as 0.34 per cent.

(7) Neither of the methods employed above seems to give concordant results in the hands of different workers. It appears that the methods are faulty.

(8) Mr. Patrick made some determinations, in which he compared the use of cold (room temperature) and hot water in washing the precipitated albumin. With both methods under discussion, he secured higher results with cold water.

Some similar determinations have been made in the Geneva laboratory on other samples of milk since receiving Mr. Patrick's results.

With the provisional method we found very little difference in the use of hot or cold water, the slight difference being in favor of hot water.

Results in washing albumin.

Treatment.	Sample A.	Sample B.
	Per cent.	*Per cent.*
Hot water (80 cc)	0.300	0.331
Cold water (80 cc)	.272	.312

With the magnesium sulphate filtrate method we found the reverse true. From special work which we have done since sending out samples it seems necessary to wash the albumin precipitate with saturated solution of magnesium sulphate if reliable results are to be secured.

Results in washing albumin.

Treatment.	Sample A.	Sample B.
	Per cent.	*Per cent.*
Washing with cold water (80 cc)	0.163	0.169
Washing with hot water (80 cc)	.119	.154
Washing with magnesium sulphate solution		.385

MODIFIED PROVISIONAL METHODS COMPARED.

In making a comparison some time ago between the provisional and modified methods for determining albumin in 45 different milks, the provisional method gave

an average of 0.315 per cent of albumin and the modification gave an average of 0.345 per cent. In 6 cases of the 45, the modification gave an average of 0.03 per cent less; in 9 cases there was no difference, and in 30 cases the modification gave more by amounts varying from 0.01 to 0.12 per cent and averaging 0.05 per cent.

In the Geneva laboratory we have in two trials with different samples obtained slightly higher results in washing with cold water.

Results in washing albumin.

Treatment.	Sample A.	Sample B.
	Per cent.	*Per cent.*
Washing with cold water (80 cc)	0.346	0.387
Washing with hot water (80 cc)	.321	.371

Determination of Water in Milk.

The phraseology employed in describing the determination of water in milk is decidedly faulty. It came about from a condensation of two methods into one without preserving the essential conditions of the two methods. It directs to heat until apparently dry and then heat for one hour at the temperature of boiling water. The phrase "apparently dry" is too indefinite. The temperature of boiling water is not necessarily high enough to complete the drying in one hour, unless we are where the boiling point is close to 100° C. When we use a steam-drying oven, as so many do, the temperature rarely reaches 100° C.

In a large number of determinations we found that when we used a steam oven it required from one and one-half to two hours to evaporate to constant weight with sand and from two to two and one-half hours or longer without sand, other conditions being uniform. The use of a water bath sometimes gave more rapid drying than the steam oven, and sometimes not.

The method should be more elastic as to time of drying and should be to constant weight rather than for a fixed time.

Methods for Detection of Adulterations in Dairy Products.

It has not been the referee's design to make any attempt at an exhaustive consideration of this subject. The number and kinds of adulterants actually used in milk and its products are comparatively few. It would be a waste of time to treat of forms of adulteration which are possible, but which are rare in actual occurrence.

An attempt has been made to give standards in a broad way. These may be open to objection as presented, but they will invite discussion and will result, it is hoped, in a consensus of opinion more important than any other that has been put on record in this country in reference to a somewhat vexatious field.

So far as it is deemed desirable, the referee has tried the tests recommended and not already a part of the association's methods, and given only those which were found to work in his hands.

Detection of Adulterations of Milk.

(1) *Forms of adulteration.*—There are three kinds of adulteration in milk which are common: First, addition of water; second, removal of fat or addition of skim milk; and, third, addition of preservatives, most prominent among these being borax, boracic acid, and formaldehyde.

(2) *Methods of detection.*—In general, it may be said that the specific gravity test by itself furnishes very little evidence in regard to the purity of milk. As regards the detection of watering or skimming, the question becomes one mainly of the inter-

pretation of results secured by a determination of fat or solids or both, in the light of a State legal standard. The chemical methods are detailed under the analysis of milk.

(*a*) DETECTION OF FORMALDEHYDE.

(1) When milk contains not less than 1 part of formaldehyde in 10,000 parts, a violet or bluish color is formed by pouring the milk into a test tube containing concentrated sulphuric acid in which there is present a trace of ferric chlorid. The color forms at the junction of the two liquids.

(2) When small quantities of formaldehyde are present, distill the milk until a distillate of several cubic centimeters is obtained. To this distillate add one drop of a very dilute aqueous solution of phenol and pour the mixture upon concentrated sulphuric acid in a test tube. A bright crimson color appears at the zone of contact. This is readily seen when the milk contains as little as 1 part of formaldehyde in 200,000 parts. If the proportion be greater than 1 to 100,000, there is above the red ring a white milky zone, and if more concentrated there will be a white or pinkish curdy precipitate.

(*b*) DETECTION OF BORAX AND BORACIC ACID.

(1) On a porcelain surface place one drop of milk, two drops of strong hydrochloric acid, and two drops of fresh, saturated, turmeric tincture (or turmeric powder). Dry on water bath; remove as soon as dry; cool, and add one drop of ammonia with a glass rod. A slaty-blue color forms, changing to green. This detects 1 part of borax in 1,000 parts of milk. When the amount is less, the test gives a green but not a blue tint.

This method may be carried out also as follows: Incinerate a few drops of milk; treat with a little water; make just acid with hydrochloric acid; add a few drops of turmeric solution, and evaporate to dryness in a porcelain dish. Moisten with dilute ammonia. A dark-blue solution forms, rapidly disappearing.

(2) Evaporate one or two drops of milk with an equal quantity of fresh, saturated, turmeric tincture. Then add one drop of strong hydrochloric acid, drawing acid over residue with a glass rod. Apply heat again for a few seconds, when, if either borax or boracic acid be present, a color is formed, ranging from pink to dark red. Cool, and confirm presence of borates by adding a drop of ammonia.

(3) *Chemical standards of purity for milk.*—The standards for pure milk vary widely in the different States. In some, fat alone is fixed; in others, total solids alone; in still others, both fat and solids are fixed; while in a few cases total solids and solids-not-fat are used. No State, except one or two, has a standard lower than 3 per cent for fat and 12 per cent total solids. In some of the countries and municipalities of Europe, the lowest limit of fat is fixed as 2.5 per cent and of total solids as 11.5 per cent. It is safe to say that while the milk of some herds of cows may occasionally fall below 3 per cent in fat, and more often below 12 per cent in solids, still milk containing less than 3 per cent of fat and 11.5 of solids at the same time is open to suspicion as having been watered. Milk in which the total nitrogen compounds (total nitrogen multiplied by 6.25) are equal to the fat may be safely regarded as having been skimmed or diluted with skimmed milk. Milk containing any form of preservative or coloring matter is in most States regarded as adulterated.

DETECTION OF ADULTERATIONS OF BUTTER.

(1) *Forms of adulteration.*—The most common adulteration of butter is substitution, in part or whole, of fat other than butter-fat. Occasionally preservatives are found, such as occur in milk. Excessive water or casein should be regarded as adulteration.

(2) *Methods of detection.*—The methods commonly employed for detecting fats foreign to pure butter are given in Bulletin No. 46, Department of Agriculture, Division of Chemistry, on pages 26 to 35.

(3) *Standard of purity for butter.*—The per cent of fat in butter should reach 80. Water should not exceed 18 per cent, and preferably should keep down to 15. Nitrogen compounds should not go much above 1 per cent.

For the purpose of determining the purity of butter-fat, the following processes are usually regarded as most helpful, their value ranking in the order given:

(1) Determination of Reichert-Meissl number—that is, the number of cubic centimeters of one-tenth normal solution required to neutralize completely the volatile acids obtained from 5 grams of fat. When this number falls below 25, the sample is open to suspicion; and when it falls to 23 or below, the sample is usually condemned.

(2) Determination of specific gravity at 40° C. When this falls below 0.91, it indicates cause for suspicion, and should be considered with other factors.

(3) Determination of the saponification equivalent or Koettstorfer's number—that is, the number of milligrams of KOH neutralized in saponifying one gram of fat. This should not be under 220.

(4) Determination of the insoluble fat acids, expressed as Hehner's number, which is the number of parts of insoluble fat acids in 100 parts of fat. In pure butter-fat this number rarely goes above 89, but has been found as high as 90.

Detection of Adulterations of Cheese.

(1) forms of adulteration.

There are only two kinds of adulteration in cheese which are common: First, the use of fat other than milk-fat, producing the so-called filled cheese; and second, the removal of fat in varying degrees, producing so-called skim cheese.

(*a*) Filled cheese is made from a mixture consisting of separator skim milk mixed in an intimate way with liquid fats or oils derived from the fat of swine and cattle. Hence, the chemical means most commonly used for detecting filled cheese are the same as are employed in detecting adulterated butter.

Since the amount of fat which can be mechanically held by skim milk is less than 3 per cent, there will be found in filled cheese a larger amount of casein in proportion to fat than is found in whole milk cheese.

(*b*) Skim milk cheese made from milk which has been skimmed enough to pay can readily be detected by the relation of fat to nitrogen compounds or to solids-not-fat, or, less certainly, by the amount of fat in cheese.

(2) methods of detection.

(1) Preparation of sample. Henzold's method, as described by Wiley, in Principles and Practice of Agricultural Analysis, is as follows: "The cheese, in quantities of about 300 grams, is cut into fragments about the size of a pea and treated with 700 cc of potash lye, which has previously been brought to a temperature of about 20°. The strength of the lye should be such that about 50 grams of the caustic potash are contained in each liter of the solution. The treatment is conveniently conducted in a wide-neck flask and the solution of the casein is promoted by vigorous shaking. After from five to ten minutes it will be found that the casein is dissolved and the fat is found swimming upon the surface of the solution in lumps. The lumps of fat are collected in as large a mass as possible by a gentle shaking to and fro. Cold water is poured into the flask until the fat is driven up into the neck, whence it is removed by means of a spoon. The fat obtained in this way is washed a few times with as little cold water as possible in order to remove the residue of potash lye which it may contain. Experience shows that the fat by this treatment is not perceptibly attacked by the potash lye. In a short time, by this procedure, the fat is practically all separated and is then easily prepared for chemical analysis by filtering and drying," as described in Bulletin No. 46, Division of Chemistry, United States Department of Agriculture, page 27, 3-(a).

The fat may be less conveniently obtained from the cheese by reducing the par-

tially dried sample to a finely divided state and applying any of the usual solvents. The solvent is removed from the extract by evaporation and the residual fat is filtered and prepared for examination as usual.

The fat may be separated also in the following manner: The cheese is ground fine by cutting into strips and passing through a meat-cutting machine. It is then placed in a flask and warm water poured upon it in the proportion of 1 cc for each gram of cheese. Agitate thoroughly. Add slowly in small quantities at a time sulphuric acid of sp. gr. 1.82 to 1.825, agitating thoroughly after each addition of acid. Use in all an amount of acid equal to the amount of water used. After standing a few minutes, the fat separates and can be removed by use of a separatory funnel, after which it can be washed free from acid and then filtered and dried.

(2) Determination of Reichert-Meissl number, Hehner's number, etc. See Bulletin No. 46, pages 26 to 35, United States Department of Agriculture, Division of Chemistry.

(*b*) *Skim-milk cheese.*—Determine the per cent of fat and of nitrogen compounds or of total solids, as directed in Bulletin No. 46, page 37, and Circular No. 2, page 3, United States Department of Agriculture, Division of Chemistry.

3. CHEMICAL STANDARDS OF PURITY FOR CHEESE.

(*a*) Filled cheese.—In respect to purity of fat in cheese, Dr. Crampton writes the referee as follows: "I have no hard and fast standard, being guided by the circumstances in each case and by the results obtained from other tests, if the Reichert-Meissl figure be low. Most of my samples, both of cheese and butter, present no difficulties. The English standard of 24 is about as good as any, in my opinion; though I would not condemn a cheese-fat or butter-fat which went below this, if it gave normal figures by other tests."

In filled cheese the proportion of fat to nitrogen compounds and to total solids falls below that of whole-milk cheese. In pure cheese, fat rarely, if ever, forms less than 50 per cent of the total solids, and in relation to the nitrogen compounds, fat rarely falls as low as 1.25 pounds for each pound of nitrogen compounds.

(*b*) Skim-milk cheese.—When the per cent of fat in cured cheese falls below 32 or 33, there is ground for suspicion that it is skim-milk cheese. But a more reliable guide is the relation of fat to total solids or to total nitrogen compounds as given above.

RECOMMENDATIONS.

The referee makes the following recommendations:

1. That the provisional method for determining casein in cow's milk be made official, inserting in line 3, page 2 (circular No. 2), after "add," the following words: "1 part of formaldehyde to 2,500 parts of milk or," and in the first line of the second paragraph substitute the word "preservatives" for "mercuric chlorid."

2. That the modified provisional method for determining albumin in cow's milk be substituted as a provisional method for the present provisional method, and that further cooperative work be done on the determination of albumin.

3. That the magnesium sulphate method for determining casein in cow's milk be made an official optional method.

4. That under milk analysis (*a*) (Bull. No. 46) the phraseology of the first and second sentences be changed to read as follows: "Heat to constant weight from 1 to 2 grams of milk in a tared flat dish, having a diameter of not less than 5 centimeters, containing from 15 to 20 grams of pure dry sand, or without sand, at the temperature of boiling water."

5. That, under the different divisions of dairy products, in the association's methods of analysis, there be added the following head and subheads: "Adulterations: (1) Forms, (2) Methods of detection, (3) Standards of purity:" and that under each be incorporated, in briefest form practicable, such statements as may hereafter be decided upon by the association.

(*c*) Determination of nitrogen compounds.

(1) Total nitrogen compounds.

Place in Kjeldahl digestion flask a known weight (about 5 grams) of milk, and proceed, without evaporation, exactly as described for this method under nitrogen, page 36, Bull. No. 46. Multiply the percentage of nitrogen by 6.25 for nitrogen compounds.

(2) Determination of casein in cow's milk by precipitation with acetic acid.

When it is not practicable to make the determination of casein in cow's milk within twenty-four hours from the time of milking, add 1 part of formaldehyde to 2,500 parts of milk, or 1 part of mercuric chlorid to 2,000 parts of milk, and keep in a cool place.

Weigh about 10 grams of milk, dilute in a beaker with about 90 cc of water at 40° to 42°, and add at once 1.5 cc of a solution containing 10 per cent of acetic acid by weight. Stir with a glass rod and let stand from three to five minutes longer. Then decant on filter, wash two or three times with cold water by decantation, and then transfer precipitate completely to filter. Wash once or twice on filter. The filtrate should be clear, or very nearly so. If the filtrate be not clear when it first runs through, it can generally be made so by two or three repeated filtrations, after which the washing of the precipitate can be completed. The washed precipitate and filter paper are then digested as in the regular Kjeldahl method for the determination of nitrogen, and the process is completed as usual. To calculate the nitrogen into an equivalent amount of casein, multiply the per cent of nitrogen by 6.25.

In working with milk which has been kept with preservatives, the acetic acid should be added in small portions, a few drops at a time, with stirring, and the addition continued until the liquid above the precipitate becomes clear, or very nearly so.

(3) Determination of casein in cow's milk by precipitation with magnesium sulphate.

Prepare a saturated solution of nitrogen-free magnesium sulphate at a temperature of 40° to 45° C. Add 50 cc of this solution to about 5 grams of milk and heat the mixture to from 40° to 45° C. until the precipitate completely separates, leaving the surrounding liquid clear. The precipitate is filtered and washed two or three times with the warm saturated solution of magnesium sulphate and then treated by the Kjeldahl method for the determination of nitrogen.

This method usually gives 0.1 to 0.2 per cent more of casein than the preceding, but the filtration is very tedious and, except when exceeding accuracy is required, the precipitation by acetic acid is sufficiently accurate for most purposes.

(4) Provisional method for the determination of albumin in cow's milk.

The filtrate obtained above in separating casein by acetic acid precipitation is just neutralized by nitrogen-free caustic alkali. Then add 0.3 cc of a 10 per cent solution of acetic acid and place in a water bath, heated to the boiling temperature of water, until the albumin separates, leaving a clear solution. The filtered and washed precipitate is then treated by the Kjeldahl method for determining nitrogen. The amount of nitrogen multiplied by 6.25 gives the amount of albumin.

(*a*) Determination of water.

(1) Evaporation in open dish.

Heat to constant weight from 1 to 2 grams of milk in a tared flat dish, having a diameter not less than 5 centimeters, containing from 15 to 20 grams of pure dry sand, or without sand, at the temperature of boiling water. Cool in a desiccator, and weigh rapidly to avoid absorption of hygroscopic moisture.

(2) Babcock asbestus method. (Unchanged.)

FOOD ADULTERATION.

By W. D. Bigelow, *Referee*, and W. S. Sweetser, *Associate Referee*.

Mr. Bigelow, referee on fermented and distilled liquors, read the following report:

At the last meeting of the association your referee was instructed to discontinue work for the present in the line of fermented and distilled beverages, and to report methods for the determination of food adulterants. It has not been found possible to include the whole subject of food adulteration, but methods have been compiled, with the collaboration of Mr. W. S. Sweetser, the associate referee, for the following classes of foods: Flour, bread, mustard, pepper, cayenne, ginger, cinnamon, cloves, allspice, nutmeg, mace, cream of tartar, baking powder, vinegar, canned foods, wine, beer, and cider. A separate chapter is also devoted to the detection of preservatives.

Flour and Meal.

Flour is a finely divided and bolted product of a single cereal. Meal is more coarsely ground, is not bolted, and in the case of barley is prepared from the unhulled grain.

The forms of adulteration to be expected are: Admixture with mineral matter to increase the weight of the flour, or with alum to improve its physical appearance; and the addition of flour or meal from the cheaper cereals. The flour may also contain seeds of weeds, such as cockle (*Lychnis githago*) and purple cow wheat (*Melampyrum arvense*); its water content may be too high; it may be more or less decomposed, or it may be made from a grain affected with fungi.

In examining flour for adulterations, the percentages of ash, fiber, ether extract, and proteids are all of value. The table below gives the extreme percentages of these compounds. It must be understood that this table is only provisional, and it will frequently happen that products which are not adulterated will not fall within these limits. At the same time it is believed that all samples of first-class flour and meal should do so.

Composition of flours and meals.

[Calculated to water-free substances.]

Flours and meals.	Moisture.		Ash.		Proteids n. × 6.25.		Fiber.		Ether extract.		Nitrogen-free extract.	
	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.
	Pr. ct.	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*
Wheat flour	15.0	9.0	0.8	0.3	15.0	8.0	1.0	0.1	2.0	0.5	90.0	82.0
Rye flour	14.0	12.0	1.5	.5	1.0	6.0	0.6	.4	1.0	.9	92.0	88.0
Buckwheat flour	18.0	12.5	1.5	.8	9.5	5.0	.6	.3	2.0	.8	93.0	84.0
Barley flour	15.0	10.0	2.0	1.0	12.0	8.5	.6	.3	2.0	.5	92.0	87.0
Pea flour	15.0	8.0	3.2	2.8	25.0	21.0	20.0	12.5	2.0	1.0	60.0	55.0
Bean flour	17.0	10.0	4.0	3.0	26.0	22.0	4.0	3.5	2.5	1.5	70.0	65.0
Vetch flour	16.0	10.0	3.5	1.6	35.0	30.0	.....	.....	2.5	1.5	.....	.....
Rice flour	15.0	10.0	.6	.3	10.0	7.0	.4	.1	0.6	.3	90.0	85.0
Potato flour	14.0	9.0	6.5	3.0	13.0	6.0	4 5	1.5	.8	.3	90.0	75.0
Graham flour	15.0	11.0	2.2	1.8	15.0	10.0	2.4	2.0	2.2	1.9	72.0	70.0
Barley meal	15.0	10.0	4.5	1.7	17.0	9.0	8.0	6.5	3.7	1.7	78.0	72.0
Oatmeal	10.0	6.0	2.4	2.0	18.0	14.0	1.4	.7	9.5	6.5	76.0	72.0
Corn (maize) meal	18.0	8.0	4.5	1.0	1.5	8.0	3.5	.7	6.0	2.5	80.0	63.0
Pearled barley meal	15.0	10.0	2.0	1.0	12.0	8.5	.6	.3	2.0	.5	87.0	92.0

Wheat flour is subjected to a far greater amount of adulteration than all other flours and meals. It will therefore be of the greatest importance to give this product especial attention. In its physical characteristics wheat flour is a white, fine powder with only the slightest yellow tinge. To judge of the color the sample should be examined in thin layers with a light not too strong. The flour should be free from acidity and its odor sweet and agreeable. When pressed with a smooth surface no bran should be revealed to the naked eye or the lens. The hand plunged into it should not experience a cooling effect. When pressed in the hand it should retain its shape for some time. A microscopic examination should reveal no foreign matter such as starches of other cereals, fungi, or weed seed. The aqueous extract should not exceed 5 per cent.

It is probable that the adulteration of flour with mineral matter, for the sake of increasing its weight, is now scarcely practiced in this country at all. It can readily be detected, however, by an abnormally high ash, and the qualitative examination of the ash will reveal the nature of the mineral used for adulteration.

DETECTION OF ALUM AND SAND.

Mix 50 grams of flour with water to form a thin paste, and then a few drops of a freshly prepared solution of logwood in alcohol. The mixture is then made alkaline with ammonium carbonate. The presence of alum will be indicated by the formation, sometimes after several hours, of a lavender-blue colored lake. Blyth prefers to immerse a strip of gelatin for twelve hours in a cold aqueous extract of the flour and subsequently in an alcoholic tincture of logwood, made alkaline with ammonium carbonate.[1] The following method is often preferable. Place 200 grams of the sample in a separatory funnel and shake with a sufficient amount of chloroform. Allow to stand overnight and carefully remove mineral matters, alum, etc., through the stopcock. The deposit may be further purified by shaking a second time with a small portion of chloroform. It is then transferred to a watch glass with as little chloroform as possible and the chloroform evaporated, the alum dissolved in water and again obtained by crystallization, the crystalline form being noted. The aqueous solution may also be tested with barium chlorid and Nessler's solution. The portion of the sediment insoluble in water may then be examined microscopically and its nature determined.

DETECTION OF FOREIGN CEREALS.

Flour which is adulterated with products manufactured from cheaper cereals presents a much more difficult field. The most satisfactory method of examining it is by means of a microscope.

A very important method for the detection of such adulterants is that for the estimation of gluten. In this determination, 10 grams of the flour are mixed with sufficient water to make a stiff dough and allowed to stand for one hour. It is then kneaded in the hand or in a piece of linen cloth in a vessel of water or, better, under a faucet, until the water which is pressed from the gluten is clear. The gluten thus prepared while fresh should have a very light yellow color—should be tough and of such consistency that it can be pulled out to a thread.

As a rule, the whiter the gluten while still in the moist state the better the quality of the flour. As the color approaches gray and red the flour becomes of a more inferior quality and less digestible, if indeed it be not adulterated with flours manufactured from other cereals. The gluten from good flour on being quickly heated to the temperature of about 150° C., swells and assumes the appearance of bread. Good flour should contain from 25 to 40 per cent of moist gluten and from 10 to 18 per cent of dry gluten. (Dried at 100° C.)

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[1] A gram of finely divided logwood chips is digested for eight or ten hours in 25 cc of alcohol, and to 10 cc of the alcoholic solution 150 cc of water and 10 cc of saturated ammonium carbonate solution are added.

Gluten obtained from a mixture of wheat and rye flour is dark and viscous, without homogeneity; from a mixture of wheat and barley flours, dark, nonviscous, or a dirty, reddish-brown color; from a mixture of wheat and oats, dark yellow; from a mixture of wheat and maize, yellowish and nonelastic.

The gluten obtained from wheat flour which has been mixed with leguminous flour varies in color from a grayish red, in the case of vetch or beans, to green in the case of peas, and has the characteristic odor and taste of leguminous products.

DETECTION OF LEGUMINOUS FLOURS.

The ash of leguminous flour is very deliquescent, is high in chlorids, and turns turmeric paper brown; cereal ash is the reverse. The aqueous extract of the leguminous flour is extremely acid, while that of cereal flour is faintly alkaline. If the filtrate from the gluten determination of flour containing leguminous flour be made alkaline with ammonia, allowed to stand overnight, and the clear liquid decanted, the addition of dilute mineral acid will precipitate legumin.

DETECTION OF POTATO FLOUR.

Rub a portion of flour vigorously in a mortar until a stiff paste is obtained, thin with more water, filter and test the clear filtrate with a drop of a dilute solution of iodin. In the presence of potato flour a deep blue color is produced, while if only wheat flour be present the resulting color is yellow or light orange. If a mixture of cereal flour and potato flour be dried, spread in a thin layer on a glazed black surface, and examined with a lens, the potato flour is indicated by bright and glassy particles in the otherwise dull white substance.

ALCOHOLIC EXTRACT.

Vogel extracts the flour with 70 per cent of alcohol, to which 5 per cent of hydrochloric acid have been added. The extract is colorless if the flour consist only of wheat or rye, pale yellow if adulterated with barley or oats, orange yellow with pea flour, purple red if made from mildewed wheat, and blood red if made from ergotized wheat.

DETECTION OF ERGOT.

The examination of flour for ergot may be preceded by a general preliminary test to determine whether the flour has been damaged by any form of fungus. Vogel directs to stain the sample of the flour with analin violet and examine with the microscope. If the starch granules have been injured by any fungus they will be found to be thoroughly stained. For the presence of ergot or similar fungi, as indicated by this test, both of the following methods should be used. If the presence of damaged starch granules be indicated by this test one of the following methods may be employed:

First method.—Twenty grams of the sample are digested with boiling alcohol as long as any color is extracted. The solution is then treated with 1 cc of sulphuric acid (1:3). The presence of ergot will color the solution red, and if the solution be diluted with quite a volume of water the red coloring matter may be extracted from separate portions by means of chloroform, ether, benzine, or amyl alcohol.

Second method.—Ten grams of the flour under examination are digested for about thirty minutes with a mixture of 20 cc of ether and 10 drops of dilute sulphuric acid (1:5); filter, and wash with ether until the filtrate amounts to 15 cc. The filtrate is then shaken with 5 drops of a saturated solution of sodium bicarbonate. The chlorophyll remains dissolved in the ether and the sodium bicarbonate solution remains clear if the flour be from sound grain, but takes on a deep violet color if ergot be present.

DETECTION OF RICE IN BUCKWHEAT FLOUR.

When pure buckwheat is mixed with water into a thin paste the addition of calcium hydroxid gives a dark green color, which becomes red when acidified with hydrochloric acid. Rice flour with the same treatment gives a yellow color with

potassium hydroxid and white with hydrochloric acid. A mixture of buckwheat and rice flours made into paste is changed to a light green color by potassium hydroxid and becomes flesh colored when acidified with hydrochloric acid

Bread.

The examination of bread for adulterants is very similar to the examination of flour. The more common adulterants are cheaper flours or meals and damaged flour. The addition of alum to bread is also frequently practiced, and instances of the addition of quite an amount of copper sulphate and zinc sulphate are not wanting. The presence of one-tenth of a gram of alum in a one-pound loaf will be detected by immersing a thin slice of bread in the alkaline logwood solution described on page 118 and allowing it to dry in the air. An evident blue color appears after from two to four hours. The presence of more than 0.03 per cent of alum may be detected by immersing a strip of gelatin for ten or twelve hours in the aqueous extract of the bread and then in the logwood solution described on page 118. The aqueous extract is made by digesting 150 grams of the dry ground bread for one or two days in two or three liters of water, then straining the mixture through a bag filter, using vacuum if necessary, and concentrating by evaporation to a small volume.

Quantitative estimation of alum.

The ash of 200 grams of bread is treated with hot hydrochloric acid, hot water added and the mixture filtered. The filtrate is treated with a strong excess of sodium hydroxid, boiled again, filtered, and washed. The filtrate thus obtained is treated with a few drops of sodium phosphate, acidified with hydrochloric acid, and the aluminum phosphate precipitated with a slight excess of ammonium hydroxid. The silica is also estimated in a separate portion of the ash. Aluminum compounds commonly occur in the ash of flour, especially in inferior flour, but probably in the form of silicate, and in these compounds the amount of silica present is approximately equal to the amount of alum corresponding to the aluminum present; therefore the weight of added alum may be obtained by subtracting the weight of silica from that of alum. One part of aluminum phosphate is equal to 3.733 parts of ammonium alum and to 4.481 parts of potassium alum.

Copper sulphate may be detected in bread by immersing a thin slice in a very dilute solution of potassium ferrocyanid, made acid with acetic acid. In the detection of foreign flours the methods given under flour and meal may be employed, but it must be remembered that the starch granules are to a large extent destroyed in the process of baking, so that their detection by means of the microscope is a very difficult matter, and with the exception of potato starch is often almost impossible.

The composition of any variety of bread should be approximately the same as that of the flour from which it is manufactured (see page 117), with the exception of the ash and ether extract. The former will be from 0.7 per cent to 1.5 per cent higher, owing to the addition of sodium chlorid, while the latter is often considerably increased by the use of lard. The amount of lard added, however, should not exceed 5 per cent of the weight of the bread. The amount of moisture in wheat bread should not exceed 45 per cent, and will more commonly be between 30 and 40 per cent. If the bread be manufactured from flour which is adulterated with potato flour or rice flour, or if mashed potatoes be added to the dough, the amount of moisture will greatly exceed these limits, and the bread will often be of a clammy consistency.

The presence of fungi and of flours which have been damaged by their action may be recognized by the methods which are given for their detection in flour.

Spices and Condiments.

Mustard.

The adulterants which are sometimes found in mustard are wheat flour, potato flour, linseed meal, gypsum, and clay, for the purpose of increasing the weight;

certain seeds, such as radish, rape, *Sinapis arvensis*, cayenne, ginger, and possibly gamboge, for the purpose of increasing weight and also of imparting something of a flavor; and turmeric and martius yellow, and possibly yellow ocher and certain chromates, for the purpose of imparting color. If the adulterant belong to the first class mentioned it will be impossible to identify it except by the use of the microscope. The presence of any flour containing much starch, however, can be detected by boiling 2 grams of the sample in a few cubic centimeters of distilled water for ten minutes, cooling and adding a few drops of iodin solution. The formation of a blue color indicates the addition of some starch or flour. Pure mustard gives no reaction with iodin.

The presence of adulterants can also frequently be proved and in many instances the extent of adulteration be shown by chemical analysis, although, except in the case of adulteration by means of mineral substances, the nature of the adulterant can not be shown. The following limits of analysis will probably include all first-class ground mustards and almost all pure ground mustards:

	Per cent.
Moisture	3 to 8
Ash	4 to 7
Ether extract	31 to 37
Fiber	4 to 6.5
Aqueous extract	30 to 38
Sulphur	1 to 1.6

It frequently happens that the ether extract will be found considerably below the limits given, since the removal of a portion of the oil is often practiced, some houses claiming that the mustard keeps better if a portion of the oil be removed. Instances are recorded also of mustards having a fiber content of from 15 to 17 per cent. A sample exceeding the limits given, however, is to be regarded with suspicion. The color of pure mustard is a very light dull yellow. Wherever a bright yellow is found, coloring matter may be expected. If yellow ocher or barium chromate have been employed, their presence may be readily detected in the ash. The most common coloring matter used in mustard, however, is turmeric. The addition of a strong solution of ammonium hydroxid will usually give an orange-red color in the presence of turmeric. If 2 or 3 grams of the sample be thoroughly mixed with castor oil and filtered, the filtrate, when turmeric is present, will show a marked fluorescence.

A more delicate and perhaps more certain test is to extract a small portion of a sample with two or three times its weight of methyl alcohol, filter, and divide the filtrate into two portions; one portion is then evaporated to dryness. If turmeric be present, the residue will be turned red by hydrochloric acid, which color will be changed to greenish blue on the addition of an excess of alkali. The other portion of the filtrate is evaporated to dryness, moistened with a solution of boric acid, and dried on the steam bath. The presence of turmeric is indicated by a cherry-red color. The addition of alkali then produces a play of colors in which green and purple predominate. Martius yellow, or naphthalene yellow (dinitro-naphthol), may be extracted by 95 per cent alcohol from the slightly acidified sample of mustard. This coloring matter dyes wool or silk (but not cotton) a bright yellow. If gamboge be present, the alcoholic extract is turned bright red by sodium hydroxid and yellow by hydrochloric acid.

Pepper.

Two forms of pepper are commonly sold—black and white pepper—both being the fruit of the *Piper nigrum*, the former being picked while green and allowed to ripen, the latter being allowed to ripen on the plant and then picked and the outer pericarp removed. The common adulterants are ground corn, wheat products, rice, pea flour, buckwheat hulls, cocoanut shells, roasted and unroasted olive stones, mustard husks, linseed meal, sawdust, and cayenne.

Unground pepper is probably not adulterated at present by the admixture of foreign

substances. Often, however, the peppercorns are much too small, and a large amount of dust and trash is present. One hundred peppercorns taken at random should not weigh less than 4 grams, and the amount of dust, stems, and trash present in black pepper should not exceed 1½ per cent.

Unground white pepper should be practically free from dust. It is said that artificial peppercorns have been made, though it is probable that there are none on the market in this country. In the examination of ground pepper, the microscope is usually necessary to identify the adulterant used. A sample should be examined in water by direct and by polarized light before and after treating with iodin. Starches should be examined and measured. Stone cells, whether their origin be olive stone or cocoanut shells, may be detected by polarized light, but great care must be taken not to confuse them with the stone cells of the outer portion of the peppercorn. In order to clear the denser tissues, a potassium hydroxid solution may be run under the cover glass after the examination in water, or a fresh portion may be digested twenty-four hours, in a solution of 8 parts of chloral hydrate in 5 parts of water, and examined microscopically in the same medium. In the preparation for the microscopical examination, a portion of the sample may be mixed with an aqueous solution of glycerol (sp. gr. 1.173), in which foreign stone cells will sink to the bottom, while the greater part of the pepper will float or remain suspended in the liquid. The sample may be bleached by allowing it to stand several hours with nitric acid (sp. gr. 1.1), adding powdered potassium chlorate from time to time. The dark-colored portion of the pepper and many vegetable adulterants will be bleached by this method, while charcoal and charred shells will retain their color. The residue from crude fiber determinations should also be examined microscopically, as usually all dark-colored matter occurring in pepper will be bleached in the course of the determinations, with the exception of charred matter. Stone cells will also show their structure best in this residue. The chemical composition of the pepper is also of considerable value in judging of the purity of the sample. The following limits of composition will probably include all good peppers:

Composition of black pepper.

	Per cent.
Moisture	8 to 12
Ash[1]	2.75 to 6.5
Starches, direct inversion by hydrochloric acid	32 to 38
Fiber[1]	9 to 16
Albuminoids	7 to 12
Nonvolatile ether extract not less than	6.5

Composition of white pepper.

	Per cent.
Moisture	12 to 15
Ash	0.8 to 2.9
Starch, direct inversion by hydrochloric acid	40 to 53
Fiber	4 to 8
Albuminoids	8 to 12
Ether extract	7 to 8

If a substance high or low in ash have been added, this will readily be detected in the analysis. The same is true of fiber and fats, while if the adulterant contain approximately the same amount of fat as is found in the pure pepper, a nitrogen determination in the extract obtained by extracting about 10 grams of the pepper with ether, will indicate the adulteration as the ether extract obtained from pepper will contain 2 to 3.5 per cent of nitrogen, owing to the presence of piperin.

[1] All samples having over 5 per cent of ash or over 11 per cent of fiber should be regarded with suspicion.

Cayenne.

The fruits of *Capsicum annuum and Capsicum fastigiatum* are ground and placed on the market under the name of cayenne, frequently called cayenne pepper, although the capsicum is not a pepper. As cayenne is sold almost entirely in the ground condition, and in this condition is just as subject to adulteration as other condiments, a very large percentage of the article in the market is adulterated.

The common adulterants which are from time to time reported are hulls of mustard and other spices, corn, rice, and wheat flour, ground stems, red sandal wood, buckwheat middlings, and occasionally mineral matter, such as red ocher, or brick dust, with turmeric as coloring matter. Sometimes, when a deeper color is desired, one of several anilin colors is employed.

The results of chemical analysis are of some importance in judging of the purity of cayenne. The following limits of analysis may be adopted:

	Per cent.
Moisture	2 to 10
Ash	5 to 10
Fiber	16 to 18
Nitrogen	1.7 to 2.2
Ether extract[1]	16 to 30
Alcohol extract	25 to 45

In order to identify the adulterant, and very often to prove that adulteration has been practiced, it is necessary to resort to microscopical examination.

Ginger.

The examination of ground ginger for adulterants presents as great difficulties as that of any other spice.

The common adulterants are exhausted ginger, mustard hulls, clove stems, cayenne, rice, wheat and potato flours, corn meal, and turmeric. The microscope may be used with some satisfaction, except in the case of turmeric and exhausted ginger. The oil cells of turmeric are so similar to those of ginger that the identification of the former as an adulterant of the latter is a very difficult matter. The presence of turmeric, however, can be detected by means of the methods given under mustard. The presence of exhausted ginger can only be shown by chemical analysis, when the percentage of volatile oil will be found too low, and the other ingredients rather high. The following extremes of composition are suggested:

	Per cent.
Moisture	9 to 12
Ash	3.8 to 7
Soluble ash	1.8 to 2.8
Fiber	2 to 8
Volatile oil[2]	1 to 2.5
Ether extract	4 to 7
Nitrogen	.9 to 1.75
Starch (direct inversion with HCl)	45 to 55

The alcohol extract should be approximately 17 per cent.

Cinnamon and Cassia.

Cinnamon is the bark of the small branches of the *Cinnamomum zeylanicum*. It is a native of Ceylon, and the greater part of it is cultivated in gardens in Ceylon. Cassia is the bark of the branches of two other species of the genus cinnamomum, and is produced largely in Bengal, China, and the Indian Archipelago.

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[1] A sample whose ether extract is less than 18 per cent is to be regarded with suspicion.

[2] A sample whose content of volatile oil is less than 1½ per cent is to be regarded with suspicion.

Cinnamon is placed on the market after the removal of the outer and inner coats. Its thickness, therefore, is from one-half to three-fourths of a millimeter. The sticks are always rolled together and consist of at least eight or ten quills, one placed inside of another. Cinnamon breaks with an uneven, somewhat fibrous fracture. Cassia, on the other hand, is a large, thick bark, the thickness usually exceeding 1½ mm, and is sometimes as high as 3 mm. Neither the outer nor inner layer in cassia is removed, and the outer corky layer is usually conspicuous. The pieces of cassia on the market never consist of one quill inside of another, as is the case with cinnamon, but are always single pieces, curved to form a crude circle. There is practically no ground cinnamon in the market. The best products that are sold for cinnamon are cassia.[1] Other adulterants are wheat and potato flours, sago meal, and the arrow root—all of which are usually baked—ground crackers, ground pease, and mineral matters, the most common form of which is probably red ocher. The volatile oil is also very commonly removed for flavoring extracts before the cinnamon is placed on the market. If a portion of ground cinnamon be boiled for a moment in water and cooled, iodin will not give a starch reaction, though an evident reaction may be obtained with cassia under the same circumstances. The composition of both cinnamon and cassia varies so widely that it is useless to adopt limits of analysis except, perhaps, in two or three items. The ash of cinnamon is from 3½ to 5 per cent, the soluble ash from 2 to 4 per cent, the volatile oil from 0.8 to 1.5 per cent. If the sample be adulterated with mineral matter this will, of course, be indicated by the high ash. If volatile oil has been removed, the small proportion of it may indicate the fact, though the natural percentage of that ingredient is so low as to render this criterion of doubtful value. With these exceptions the microscope must be used for the purpose of detecting adulteration, and the appearance of cinnamon and cassia, as well as of the substances that are used for their adulteration, is so characteristic that no difficulty is encountered in the microscopical examination.

Cloves.

Unground cloves should consist of both calyx and corolla, the latter consisting of four petals closely imbricated into a globular bud. The calyx is a dark-brown color and the corolla is somewhat lighter. On scratching the corolla with a pin the volatile oil appears. When the oil has been removed the clove becomes almost black and its surface wrinkled and blistered.

Ground cloves are probably rarely sold in a pure state. The common adulterants are clove stems, allspice, flour, and burnt shells, as well as the adulterants that are used with other spices. The volatile oil is also frequently removed and the exhausted cloves put on the market either by themselves or after mixing with unexhausted samples. The limits of composition of cloves are:

	Per cent.
Mustard	3 to 10
Ash	5 to 13
Volatile oil	10 to 20
Non volatile ether extract	7 to 11
Fiber	6.5 to 9
Nitrogen	0.8 to 1.2
Oxygen equivalent	4 to 6

Tannin equivalent, 11 to 14 cc of tenth normal potassium permanganate.

The determinations which are of value in detecting the adulteration of cloves are volatile oil, fiber, nitrogen, ash, and tannin or oxygen equivalent. For the determination of the latter, 4 grams of the sample, previously extracted with absolute,

[1] The sale of ground cassia under the name of cinnamon should not constitute a form of adulteration, but "ground cinnamon" should be understood to mean the bark of any species of cinnamonum.

alcohol-free ether, are placed in a 100 cc flask with 80 or 90 cc of water and digested over night at a temperature of from 90° to 95°. The mixture is then cooled to room temperature, diluted with water to 100 cc, thoroughly mixed by shaking, filtered, and titrated with a tenth normal solution of potassium permanganate, according to directions given in Bulletin 46, Division of Chemistry, United States Department of Agriculture, page 74.

Allspice or Piment.

In flavor and composition, allspice closely resembles cloves, as would be expected from the fact that they are botanically very closely related. Being among the cheapest of the spices, however, it is not subject to such universal adulteration as is the case with many of the others. The adulterants commonly used are cocoanut shells, sandal wood, mustard hulls, ground husks, and clove stems, as well as those employed with other spices, and the volatile oil is frequently removed before the spice is placed upon the market. The limits of analysis are:

	Per cent.
Moisture	5.5 to 12
Ash	3 to 5
Volatile oil	2 to 5
Ether extract	7 to 13
Fiber	13 to 22

Tannin equivalent 5 to 7 cc of tenth normal potassium permanganate. The tannin is estimated according to the directions given under cloves.

Nutmeg.

Of all the spices nutmeg is probably the least subject to adulteration, since it is sold almost entirely in the unground state. It is said that artificial nutmegs have been prepared from mace, starchy material, and clay, or some other mineral matter, with the addition of a little nutmeg oil. If such imitations are really on the market they can readily be detected by the appearace of the cross section as viewed by the naked eye. The cross section of nutmeg has a marbled appearance, caused by narrow strips of the inner seed coat penetrating to the center of the seed.

Mace.

Mace is most commonly adulterated with what is known as "false" or Bombay mace. Other adulterants are turmeric and mineral substances, such as ocher and baryta. The microscopical appearance of mace is characteristic, and it requires but little practice for the observer to familiarize himself with the powder sufficiently to detect adulterants.

Bombay mace is very similar in structure to the true mace, but has a coarser structure, is of a much darker color, is odorless and tasteless. It is also said to be much higher in oil and ash, and its presence, as well as that of the other adulterants mentioned, may be detected from the chemical analysis. The limits of composition of mace are:

	Per cent.
Moisture	4.5 to 12
Ash	2 to 4
Volatile oil	5 to 9
Ether extract	30 to 40
Crude fiber	4.5 to 10
Nitrogen	0.7 to 1

Mace, like nutmeg, contains no fat. The extract, other than nonvolatile oil, consists almost entirely of resin. The presence of an adulterant containing less than 20 per cent of resin can be easily detected by the fact that true mace contains from 20 to 35 per cent of that substance.

The most common adulterations met with in this substance are calcium sulphate, calcium carbonate, alum, acid phosphate of calcium, and starch. Calcium tartrate is found in nearly all tartars, but this can not be considered an adulterant, since its source is the argol from which the cream of tartar is manufactured. This impurity, however, should not exceed 10 per cent.

Qualitative examination.—This may be made as follows:

Free tartaric acid is tested for by treating a portion of the sample with alcohol. The filtrate from this is evaporated free from the alcohol and a test made for acid. This may be made a quantitative determination by titrating against a standard alkali solution.

Test for *phosphoric acid* in a nitric acid solution by adding ammonium molybdate solution. If sulphates be present, a hydrochloric acid solution of the ash should be tested for *aluminum*. If this be found, a test for ammonia in the original substance gives additional evidence of the presence of alum.

Earthy additions are left undissolved after treatment with a warm potassium hydroxid solution.

The washed residue is examined for carbonates, sulphates, calcium, barium, silica, etc. This operation may be made a quantitative one by drying and weighing the residues; if calcium sulphate be present, alcohol is added in considerable quantity before filtering and in the washing.

Starch.—From 2 to 5 grams of the substance are weighed into an Erlenmeyer flask and 150 to 200 cc of a 4 per cent hydrochloric acid solution added. The flask is provided with a stopper, and condensing tube 3 to 4 feet long. Boil gently for four hours, cool, neutralize with sodium hydrate, and make up to a definite volume. Determine the reducing power of an aliquot portion by methods given in Bulletin No. 46, Division of Chemistry, United States Department of Agriculture.

Phosphoric acid, aluminum, and calcium.—Weigh out 2 grams of the sample, incinerate, transfer to a 200 cc flask, and dissolve by boiling in 30 cc of nitric acid. Make up to the mark, and estimate phosphoric acid as in fertilizer analysis. Slightly alkalize the filtrate from the ammonium phosphomolybdate with ammonia, to precipitate the aluminum as hydrate. Heat and long standing should be avoided, else the molybdic acid will be precipitated. Filter, wash, ignite, and weigh as aluminum oxid. The calcium is precipitated from the last filtrate as the oxalate, ignited and weighed as the oxid. Or, the calcium may be determined by evaporating an aliquot part of the original nitric acid solution to a very small volume, with a little sulphuric acid, and then adding two to three times the volume of 95 per cent alcohol. Filter by means of a Gooch crucible, wash with alcohol, ignite a few seconds at low red heat, and weigh as calcium sulphate.

Total tartaric acid.—Weigh out 5 grams of the material and transfer to a 500 cc flask. Add 100 cc of water and 15 cc of strong hydrochloric acid. Make up to the mark and allow to settle. To 50 cc of the filtered solution add 10 cc of a solution containing 300 grams of potassium carbonate to the liter. Boil thirty minutes, filter into a porcelain dish, and evaporate to 10 cc. Add gradually, with constant stirring, 4 cc of glacial acetic acid and then 100 cc of 95 per cent alcohol, stirring until the precipitate appears crystalline. Allow to stand several hours, until the precipitate settles well, decant through a small filter, add alcohol, and wash the precipitate on the filter with alcohol until free from acetic acid. Transfer the filter and precipitate to a beaker, add water, and boil, washing out the dish with boiling water if any of the precipitate adhere to it. The resulting solution is titrated against a decinormal soda or potash solution, using phenolphthalein as an indicator. One cubic centimeter of the decinormal alkali corresponds to 0.0188 gram of potassium bitartrate or 0.015 gram of tartaric acid.

Calcium tartrate.—In the presence of other calcium salts, the powdered tartar is treated with warm potassium hydroxid solution, adding alcohol if the sulphate be present. The residue is washed on a filter, the filtrate neutralized with hydrochloric

acid, and evaporated to a small volume. The calcium is precipitated with ammonium oxalate in the presence of a little free acetic acid. The calcium oxalate is collected and washed in a Gooch crucible and weighed as calcium carbonate, after gentle ignition, or as calcium oxid after complete ignition.

Baking Powders.

These consist of bicarbonate of soda with some acidifying agent, usually bitartrate of potassium, acid phosphate of calcium, or alum. Tartaric acid is occasionally used for this purpose. These agents are mixed with some substance called "filling," which is usually starch, although flour is used sometimes. This filling checks deterioration in the strength of the powder, by preventing the acid and alkali from acting upon each other readily. Ammonium carbonate is sometimes used to increase the strength of the powder.

The impurities most commonly met with are calcium sulphate, calcium tartrate, and insoluble calcium phosphate. These are not called adulterations, since they are introduced with the ingredients of the powder.

QUALITATIVE TESTS.

Sulphates.—Sulphates are detected in an aqueous solution, in the absence of citric acid, by treating with barium chlorid and hydrochloric acid.

Ammonia salts.—Ammonia salts are detected in the cold-water extract by boiling with sodium hydroxid and testing the steam with moist, red litmus paper.

Phosphates.—Dissolve the ash in nitric acid and add ammonium nitrate and ammonium molybdate solution. Test for insoluble phosphates by boiling the powder with water. Then filter, wash, and dissolve the residue with hot nitric acid. Test solution for phosphates as above, page 126.

Alumina.—Dissolve the powder in very dilute nitric acid, add acetic acid and sodium phosphate. Or, fuse with potassium nitrate and sodium carbonate, extract with boiling water, and filter. Acidify the filtrate with hydrochloric acid, and precipitate the alumina with ammonia.

Tartaric acid and tartrates.—Free tartaric acid is dissolved out by absolute alcohol and identified. Tartrates are tested for by shaking the powder in ammonia, filtering, adding a crystal of silver nitrate, and gently heating to form a silver mirror.

It should be noted whether there be any considerable amount of residue insoluble in boiling water other than starch. If such be the case, its nature should be ascertained.

QUANTITATIVE DETERMINATIONS.

Available carbonic acid.—This is the most important estimation, as it determines the leavening strength of the powder. Place 1 gram of the sample in a dry alkalimeter containing 10 cc of water in the acid bulb. This is allowed to run into the powder slowly, after which it is gradually brought to the boiling point. Then the lamp is removed and the apparatus is aspirated thoroughly.

Total carbonic acid is determined in the same manner, except that 10 cc of dilute sulphuric acid are placed in the bulb instead of pure water.

Starch, phosphoric acid, tartaric acid, aluminum, and *calcium* are estimated the same as in the cream of tartar analysis. In case of phosphoric acid it is well to incinerate before dissolving in nitric acid.

Sulphuric acid.—Weigh 0.5 to 1 gram of the sample into a beaker and digest with strong hydrochloric acid until all goes into solution. Add barium chlorid, in slight excess, while still hot. Allow to stand twelve hours, filter, ignite, and weigh.

Potash and soda.—Weigh out 5 grams of the substance into a dish and incinerate at low heat. It is safer to add an excess of sulphuric acid before incinerating. With the aid of a little hydrochloric acid, transfer to a 500 cc flask, add water, and boil fifteen minutes. When cool, make up to the mark. One hundred cubic centimeters

of the filtered solution are heated on a water bath, a slight excess of barium chlorid added, and then, without filtering, a slight excess of barium hydroxid. The precipitate is filtered off and washed. To the filtrate is added a little ammonium hydrate, and then ammonium carbonate until all the barium is precipitated. The precipitate is filtered off and washed and the filtrate is evaporated to dryness in a platinum dish, after acidifying with dilute sulphuric acid. The residue is thoroughly ignited and weighed as mixed sulphates of potassium and sodium. The potash is then determined in the residue by dissolving in hot water and hydrochloric acid, adding platinum chlorid, and proceeding in the usual way.

Ammonia is present either as the carbonate or as ammonia alum. It may be determined by distilling 1 gram of the sample with caustic soda. The distillate is received into an excess of standard acid solution and titrated back with a standard alkali solution. In case flour is used as a filling, which is not often, it must be separated from the ammonia salts by washing with water, and the ammonia determined in the filtrate.

A water solution of a baking powder should be about neutral; but in poorly made powders it is often alkaline. If such be the case, the free alkali should be determined by titration.

Vinegar.

The adulterations most frequently met with in vinegar are water, pyroligneous acid, mineral acids, usually sulphuric or hydrochloric, caramel, and other coloring matters.

Cider vinegar usually contains from 3.5 to 6 per cent acetic acid, 1.5 to 2.5 per cent solids, and has a density usually given from 1.013 to 1.016 at 15° C., but it often goes far beyond these limits. The dried residue has the odor of baked apples, contains malic but no tartaric acid, and gives slight precipitates with barium chlorid, silver nitrate, and ammonium oxalate.

Wine vinegars run high in acetic acid and density, and have from 1.7 to 2.5 per cent solids. The extract, treated with alcohol, leaves a granular residue of acid tartrate of potassium. The average amount of this salt is about 0.25 per cent. Its presence is peculiar to wine vinegars.

Beer and *malt vinegars* have a high density, ranging from 1.021 to 1.025, and a high percentage of solids, from 5 to 6 per cent. They also contain a high percentage of phosphates, and considerable quantities of sulphates and chlorids.

The specific gravity is determined by a Westphal balance, or a picnometer, at a temperature of 15° C.

The acidity is determined by titrating against a decinormal sodium hydroxid solution, using phenolphthalein as indicator. The result is calculated to percentage of acetic acid.

Free mineral acids.—A solution of logwood is prepared by pouring 200 cc of boiling water on 2 grams of logwood chips and allowing to stand for a few hours before filtering. Dry a drop of this solution on a porcelain surface, add a drop of the vinegar to the residue and dry again. The presence of a mineral acid imparts a red color to the residue. If the proportion of mineral acid be very small, the red color is destroyed by the addition of water, but is restored on reevaporation unless nitric acid is present. If mineral acids be present, add enough sodium hydroxid solution to 50 grams of the solution to neutralize the mineral acids, but not enough to render the liquid alkaline. Evaporate and carefully ignite. Titrate back with standard acid. The difference in the amounts of alkali and acid used is the amount neutralized by the free mineral acids.

Free sulphuric acid.—Evaporate 100 grams of the sample to a small volume; cool; add four to five times its volume of alcohol. Sulphates are precipitated and free sulphuric acid remains in solution. Filter; wash the precipitate with 80 per cent alcohol; boil off the alcohol from the filtrate after dilution, and precipitate the sulphuric acid as barium sulphate.

Solids.—Evaporate 10 grams of the sample to dryness on a steam bath and then dry to a constant weight at 100° C.

Ash.—Burn solid residue at dull red heat. Test the ash, after moistening, with litmus. If it be neutral, free mineral acids are probably present in the sample. If it be alkaline none can be present.

Phosphoric acid.—Dissolve the ash in nitric acid, filter, and add a few crystals of ammonium nitrate. When these are dissolved, add about 25 cc of the clear ammonium molybdate solution used in fertilizer analysis. Digest, with occasional stirring, for thirty minutes in a warm place, not allowing the temperature to rise above 65° C. Let stand one hour in the cold and filter through a weighed Gooch crucible. Wash with water slightly acidulated with nitric acid. Dry thirty minutes at 105° to 110° C. and weigh. Calculate the percentage of phosphoric acid by the use of the factor 0.0367.

Canned Food.

The practice of preserving various kinds of food in hermetically sealed receptacles has become so common that far more attention should be given to canned food which is on the market than it receives at the present time, and methods for its examination should be adopted. The appearance of the cans themselves should first be noted. The ends should be slightly concave, since the contents are sterilized by means of heat and a contraction of volume must necessarily follow cooling. If the ends of the can be convex, some decomposition is indicated. In the case of bottled goods, the pressure on the cork should be observed since even the slightest pressure tending to expel the cork is an indication that some decomposition has taken place after the goods were put up. Marked corrosion on the inside of the can is often an indication of the presence of metals in solution. No food which is capable of corroding metal receptacles should be preserved in them. In canned corn, corrosion may be sometimes due to the presence of sodium sulphite, which is added for the purpose of bleaching the corn. In such cases dark nodules of tin sulphid are also found at the edge of the can.

Any sample of canned goods which has an alkaline reaction with litmus paper is to be regarded with suspicion.

Among the various classes into which canned food may be divided are meats, meat extracts, fruits, vegetables, and catsups. The most common adulterants, perhaps, are added for the purpose of better preserving the food, and are treated of in some detail under the head of preservatives. To prepare a sample of canned food in such a manner that the methods given for detection of preservatives may be applicable, from 50 to 200 grams of the sample, previously ground, if necessary, are mixed in a mortar with enough water to give the proper degree of fluidity.

Coloring matter is also very commonly used, especially with food of a marked red or green color, such as tomatoes, catsups, some varieties of meat, peas, beans, and pickles. The salts of several metals are often present, sometimes having been added intentionally, at other times due to carelessness or the use of improper receptacles. Among these metals the most important are copper, lead, tin, and zinc. A slight amount of copper may be normal to many vegetables. A somewhat larger amount may come from the use of copper utensils; but if the utensils be kept clean, and ordinary care exercised in the preparation of the food, only a slight amount of the metal will be introduced in this way. The presence of more than 10 mg of copper per kilogram of the food may be taken as sufficient evidence that copper has been intentionally added. This practice is only resorted to for the purpose of giving a marked green color to foods to which a portion of the public has been educated to ascribe a green of unnatural brightness, especially peas, beans, and cucumber pickles. Zinc has in a few cases been added for the same purpose, but its use as an agent in preserving the coloring matter has not become at all general. Its presence in canned food is very common, but is almost always due to its use as a flux in soldering.

Lead comes from the use of inferior tin plate or, more commonly, solders which are much too soft. The tin plate employed should be almost free from lead, and solder which is exposed to the contents of the can should contain less than 10 per cent of lead. Owing to the lack of legislation on the subject in this country, however, the solders commonly used by our canners contain from 40 to 60 per cent of lead, and not only is a large amount of this solder exposed to the contents of the can in the seams, but it often happens that one or more pellets of the size of bullets are found loose in the can.

Tin is frequently dissolved from the tin plate by strongly acid foods. It is probable that the amount of tin sulphid is much greater where the soldering or plate exposed to the contents of the can, contains a large percentage of lead. It has been suggested that this is due to electrolytic action.

In examining a sample of canned goods for heavy metals, it should first be dried at the temperature of boiling water and charred in a porcelain dish at the lowest possible temperature. The soluble compounds may then be removed by washing the char with a few cubic centimeters of dilute nitric acid and filtering. The char and filter are then ignited, and the residue, dissolved in a little dilute nitric acid, added to the filtrate from the char. The ordinary qualitative and quantitative methods for the metals enumerated may then be applied to the solution.

Wines.

The practice of adulterating wines is so common and the methods of adulteration have been studied so long and so carefully that the detection of the adulterants used is often attended with the greatest difficulty.

The composition of dry wines and sweet wines, calculated to sugar-free substances, should not vary far from the following figures:

Composition of wines.

Constituents.	Grams per 100 cc.	
	Red.	White.
Alcohol, by volume	12.0	11.50
Extract, less sugar	2.80	2.00
Total acidity	.50	.55
Tannin and coloring matter	.25	.96
Ash	.28	.20

These figures are for young wines. After standing two or three years, the percentage of extract will decrease about 10 per cent. Wines should be regarded with suspicion which contain less than 2.4 grams of extract or more than 3.2 grams per 100 cc, or which contain less than 0.2 gram of ash or more than 0.35 gram per 100 cc, whose total acidity is less than 0.45 gram HCl per 100 cc, whose ash is less than one-eighth or more than one-half of the sugar-free extract, whose sulphuric acid calculated to potassium sulphate is over 0.2 gram per 100 cc, and whose chlorin calculated to sodium chlorid is over 0.2 gram per 100 cc.

With American wines, the presence of volatile acid, calculated to acetic acid, in excess of one-fourth of the total acid may be considered almost certain evidence of adulteration, though with European wines the ratio is somewhat larger. Wines sometimes contain an appreciable amount of copper, lead, and zinc, and sometimes, though rarely, traces of arsenic are found, owing to the use of crude glucose and anilin dyes for the purpose of coloring. Copper, lead, and zinc may be detected as directed under canned foods. For the detection of arsenic, the extract should be digested with 2 cc of sulphuric acid and about 25 cc of nitric acid to destroy the organic matter, heated with sulphuric acid until nitric fumes are entirely expelled, and the residue examined with the Marsh apparatus.

Methods for the detection of preservatives are given on page 132.

DETECTION OF COLORING MATTER.

First method.—Add 0.2 gram of precipitated mercuric oxid to 10 cc of wine; shake for one minute and filter.

Pure wines give filtrates which are colorless or light yellow, while the presence of a more or less red coloration indicates that an anilin color has been added to the wine.

Second method.—Evaporate the alcohol from 200 cc of wine, add 2 to 4 cc of a 10 per cent solution of hydrochloric acid, immerse some threads of fat-free wool, and boil for five minutes. Remove the threads, wash them with cold water acidified with hydrochloric acid, then with hot water acidified with hydrochloric acid, then with pure water, and dissolve the color in a boiling mixture of 50 cc of water and 2 cc of concentrated ammonia. Replace the threads with new ones, acidify with hydrochloric acid, and boil again for five minutes. In the presence of anilin colors to the amount of 2 mg per liter, the threads are dyed as follows:

Safranin	light rose red.
Vinolin	rose red to violet.
Bordeaux red	rose red to violet.
Ponceau red	rose red.
Fuchsin	dirty white.
Tropæolin 00	straw yellow.
Tropæolin 000	light orange.
Corallin	dirty white.

This method is not suitable for the detection of fuchsin or corallin.

Detection of fuchsin and orseille.—To 20 cc of wine add 10 cc of lead acetate solution, heat slightly, and mix by shaking. Filter into a test tube, add 2 cc of amyl alcohol and shake. If the amyl alcohol be colored red, separate it and divide it into two portions. To one add hydrochloric acid, to the other ammonia. When the color is due to fuchsin, the amyl alcohol will in both cases be decolorized; when due to orseille, the ammonia will change the color of the amyl alcohol to purple violet.

Beer.

Beer is made by fermenting the aqueous extract of malted barley, after the addition of hops. In some countries, there is legislation providing that beer shall be manufactured exclusively from malt, hops, and water. In this country, however, the use of certain malt substitutes is considered legitimate. If unmalted barley be employed as a malt substitute, or any other cereal whose content of insoluble albuminoids is equal to that of malt, the food value of the beer is probably not decreased. If dextrin, starch, or rice be employed, however, the content of albuminoids will be materially reduced. The important determinations for judging of the quality of the beer or of its adulteration are alcohol, specific gravity, glycerin, ash, sulphuric acid, sulphurous acid, phosphoric acid, and chlorin. It is also essential to examine qualitatively for the presence of preserving agents and saccharin. The methods for the detection of the presence of the last two named are given under appropriate headings. Hydrochloric acid may be determined in the nitric-acid solution of the ash, by any of the well-known gravimetric or volumetric methods. The other substances named may be determined by the methods given under the adulteration of wine. The glycerol content of beer should not be less than 0.1 gram per 100 cc. The ash should not be less than 0.12 nor greater than 0.30 gram per 100 cc. The presence of less than 0.10 gram indicates that some malt substitute which is low in ash, such as starch, has been used in the preparation of the beer, while if the ash content be greater than 0.30 gram per 100 cc, and the volatile acids, calculated to acetic acid, less than 0.075 gram per 100 cc, it is probable that an excess of acid has been neutralized by sodium carbonate, and the ash of the beer should be examined for both sodium and carbonic acid. The presence of phosphoric acid should not be less

than 0.05 gram nor greater than 0.10 gram per 100 cc. If less than 0.05 gram, it is probable that a portion of the malt has been replaced by starch or some similar substance. In European countries, hop substitutes are said to be frequently employed. Their presence has never been noticed in American beers, however, and the low price of hops makes it entirely improbable that substitutes are to be found in American beers.

Cider.

Cider is the juice of the apple, either fresh or partially or wholly fermented, but without the addition of any other substance. The composition of cider is—

Composition of cider.

Constituents.	Grams per 100 cc.
Alcohol	Below 7
Extract	2 to 8
Sugar	0.4 to 4
Total acidity, calculated to H_2SO_4	0.2 to 0.5
Fixed acids, calculated to H_2SO_4	0.1 to 0.27
Ash	0.17 to 0.35
Potassium carbonate	0.14 to 0.23

The adulterations to which cider are subjected in this country are dilution with water and the addition of alkali to correct acid fermentation, and especially the addition of preservatives. The percentage of alcohol increased by 49 per cent of the content of sugar and 97 per cent of the content of volatile acid, calculated to acetic acid, should not be less than 3 nor greater than 9. The percentage of extract should be from 2 to 8, including sugar, and obviously, if the percentage of alcohol be high the percentage of sugar will be correspondingly low. The presence of less than 1.7 per cent of ash may be taken as evidence of dilution with water, while if the percentage of ash be greater than 3.5 it is probable that a carbonate has been added for the purpose of neutralization.

Preservatives.

The methods given below for the detection of preservatives are applicable to liquids only. If it be desired to apply them to solid or semisolid substances, it will be necessary to grind these in a mortar with water until a sufficient degree of fluidity is obtained. It may sometimes be necessary to macerate the substance in some way before it can be ground in the mortar.

Salicylic acid.

First method.—Shake about 100 grams of the sample, after adding 5 cc of dilute (1:3) sulphuric acid, with 25 cc of chloroform. Allow to stand over night, remove the clear filtrate (not the emulsion) by means of a pipette, and evaporate to dryness at a low temperature. Take up with 2 or 3 cc of water, filter into a clean porcelain dish, and test with a few drops of a 0.5 per cent solution of ferric chlorid. The formation of a marked purple or violet color is sufficient evidence of the presence of salicylic acid.

Second method.—Acidify 100 cc of the liquor with sulphuric acid and extract with sulphuric ether. Evaporate the extract to dryness, warm the residue carefully with 1 drop of concentrated nitric acid, and add 2 or 3 drops of ammonia. The presence of salicylic acid in the liquor is indicated by the formation of the yellow color of ammonium picrate, and may be confirmed by dyeing a thread of fat-free wool.

Third method.—Place 100 cc of the wine in a separatory funnel, add 5 cc of sulphuric acid (1:3), and extract with a sufficient quantity of a mixture of eight or nine

parts of ether to one part of petroleum ether. Throw away the aqueous solution, wash the ether once with water, then shake thoroughly with about 50 cc of water, to which from 6 to 8 drops of 0.5 per cent solution of ferric chlorid have been added. Discard the aqueous solution, which contains the greater part of the tannin in combination with iron, wash again with water, transfer the ethereal solution to a porcelain dish, and allow to evaporate spontaneously. Then heat the dish on the steam bath, take up with 1 or 2 cc of cold water, filter into a test tube, and add 1 or 2 drops of 0.5 per cent solution of ferric chlorid. The presence of salicylic acid is indicated by the appearance of a violet-red coloration. In the case of red wines a second extraction of the residue with ether mixture is sometimes necessary. This is determined by the color of the residue left in the dish on the evaporation of the ether.

This method can not be used in the examination of beers and ales.

Fourth (Girard's) method.—Extract a portion of the acidified liquor with ether as in the preceding methods, evaporate the extract to dryness, and extract the residue with petroleum ether. The residue from the petroleum ether extract is dissolved in water and treated with a few drops of a very dilute solution of ferric chlorid. The presence of salicylic acid is indicated by the appearance of a violet-red coloration.

BENZOIC ACID.

First method.—Benzoic acid is used for the preservation of a large variety of foods. It is probably the preservative most commonly used with catsups and preserves.

Extract from 100 to 200 grams of the sample by any of the methods given under salicylic acid, evaporate the ether or chloroform extract to dryness, take up the residue with 8 or 10 cc of water, filter, neutralizing the filtrate as exactly as possible with a dilute solution of sodium hydroxid, being careful not to add the sodium hydroxid in excess, and treat with a few drops of a 0.5 per cent solution of ferric chlorid. The presence of benzoic acid will be indicated by the formation of a voluminous brownish-colored precipitate of ferric benzoate.

Second method.—Five hundred cubic centimeters of the sample are distilled almost to dryness after the addition of 20 cc of sirupy phosphoric acid. The distillate is then extracted with several successive portions of ether and the ethereal solution treated by Mohler's method.

The details are as follows: Evaporate off the ether from the resulting extract, and treat the residue with 2 or 3 cc of strong sulphuric acid. Heat till white fumes appear. Organic matter is charred and benzoic acid is converted into sulphobenzoic acid. A few crystals of potassium nitrate are then added. This causes the formation of metadinitrobenzoic acid. When cool the acid is poured into water and ammonia added in excess, followed by a drop or two of ammonium sulphid. The nitro compound becomes converted into ammonium metadiamidobenzoate, which possesses a peculiar reddish-brown color. This reaction takes place immediately and is seen at the surface of the liquid without stirring. Salicylic acid will sometimes give the same reaction, but only after waiting some minutes. The benzoic acid must first be separated in a state of approximate purity before this test can be applied. Half a milligram of the acid can be detected in the absence of interfering bodies.

FLUORIDS.

The use of fluorids as antiseptics is now becoming quite common, especially for the preservation of wines. One hundred grams of the sample, made slightly alkaline with ammonium carbonate, are heated to boiling and the fluorin precipitated with a few cubic centimeters of calcium chlorid solution. The boiling is continued for five minutes after the precipitation, the precipitate removed by filtration, washed with a little water, dried, and ignited in a small platinum crucible. One cubic centimeter of strong sulphuric acid is added, and the crucible is covered with

a watch glass coated with beeswax (marked in such a way as to permit it to be etched at some point) heated on a water bath for an hour at a temperature of from 75° to 80°. One mg per kg can be readily detected by this method.

BORIC ACID.

First method.—Boric acid and borax have long been used for the preservation of many articles of food, especially milk and meats. A great many preservatives are placed on the market which consist especially of boric acid, either free or combined. For the detection of boric acid, from 50 to 100 grams of the sample are made alkaline with sodium hydroxid or lime, dried, and thoroughly ignited. The ash is acidified with hydrochloric acid, a drop of turmeric tincture added, and the dish placed on the steam bath. The presence of boric acid imparts a cherry-red color which is unmistakable, even when present in very small quantities. It is best, however, to always conduct a blank experiment, as a pale-brown color is commonly given by the reagents themselves.

Second method.—Another method is to make 100 grams of the sample alkaline with sodium hydroxid or lime, evaporate to dryness, and incinerate as given above. Digest the residue in a few drops of dilute sulphuric acid, add 3 or 4 cc of alcohol, transfer the mixture to a platinum crucible, placing the cover in such a position that a small opening is left, and heat gradually and gently until the alcohol distills and may be burned from the opening that is left at the top of the crucible. The presence of boric acid is indicated by a green flame. If sufficient care has been exercised in the distillation, the flame will be free from the influence of sodium or calcium, and the green boric-acid flame may be recognized even if but traces of the substance be present.

ALKALINE FLUOBORATES AND FLUOSILICATES.

These preservatives are frequently overlooked in the examination of foods, as at low temperature they are decomposed, volatile compounds being formed. For their detection 200 grams of the sample are made alkaline with lime water, evaporated to dryness, and incinerated. The ash is treated with water slightly acidified with acetic acid and filtered. The filtrate may be examined as directed under "Boric acid" and the precipitate as directed under "Fluorids."

PHENOL DERIVATIVES.

Several compounds closely related to and derived from phenol and naphthol have been used for the preservation of fats to some extent, although their use is by no means common. Two hundred grams of the sample are acidified with dilute sulphuric acid and distilled with steam. The first 200 cc of the distillate are extracted in a separatory funnel with 15 or 20 cc of chloroform. The chloroform is separated, rendered slightly alkaline with potassium hydroxid, and heated almost to boiling for a few minutes. The presence of a compound belonging to the class mentioned will be shown by a marked color. Phenol becomes light red, changes to brown, then bright yellow, and finally colorless. O-cresol takes on a lilac color with a tinge of orange. β-naphthol gives a deep Prussian blue, changing to green, and finally to brown. Salol takes on a light-red color.

SULPHITES.

Sulphites are very often added to wine for the double purpose of bleaching and preserving. Since the sulphuring of the casks is a common practice and is allowable, it is a very difficult matter and often impossible to tell whether the sulphurous acid is due to the sulphuring of the cask or to the addition of a sulphite. The only way to control the matter is to prescribe an allowable limit for the amount of

sulphurous acid present. It may be detected as follows, and should not exceed 80 mg per liter:

One hundred cubic centimeters of wine are distilled in a current of carbon dioxid, after the addition of phosphoric acid, until about 50 cc have passed over. The distillate is collected in accurately standardized iodin solution. When the distillation is finished, the excess of iodin is determined with standardized sodium thiosulphate solution, and the sulphurous acid calculated from the iodin used.

ABRASTOL OR ASAPROL.

Abrastol has been suggested for the preservation of food, and is said to have been used to some extent for this purpose in Europe. Its use in this country has not become general, however, and it is quite probable that it does not occur in American foods at all. Bellier's method is probably the most delicate that has yet been suggested for its detection.

Fifty cubic centimeters of the sample are made alkaline with a few drops of ammonia, gently shaken for two minutes with 10 cc of amyl alcohol (alcohol is added if an emulsion be formed), the amyl alcohol is decanted, filtered if turbid, and evaporated to dryness. The residue is thoroughly moistened with 2 cc of a mixture of equal parts of strong nitric acid and water, heated on the water bath until half of the water is evaporated, and transferred to a test tube with the addition of 1 cc of water. About 0.2 gram of ferrous sulphate are now added, and then an excess of ammonia, drop by drop, with constant shaking. If the resulting precipitate be of a reddish color, it is dissolved in a few drops of sulphuric acid, and ferrous sulphate and ammonia are added as before. As soon as a dark-colored or greenish precipitate has been obtained 5 cc of alcohol are added, the precipitate is dissolved in sulphuric acid, and the fluid is well shaken and filtered In the absence of abrastol this method gives a colorless or light-yellow liquid, while a red color is produced in the presence of 0.01 gram of abrastol. The author states that the method is applicable to the examination of wine, beer, sirups, and preserved food.

Fats should first be melted, extracted with hot 20 per cent alcohol, the filtered alcohol evaporated to dryness, and the residue treated as given above. Salicylic acid gives a more orange-colored liquid when treated in the same manner, and when this liquid is treated with a few drops of a very dilute ferric chlorid solution the well-known violet reaction is produced, while abrastol gives a blue color with ferric chlorid, which disappears on heating. The natural coloring matter is not extracted from the alkaline liquid by amyl alcohol, and anilin colors may be removed by evaporating to dryness with wool, in the presence of acetic acid or ammonium hydroxid, according to the nature of the coloring matter, when water will dissolve only the abrastol.

FORMALDEHYDE.

Distill about 100 grams of the prepared sample until the distillate measures about 20 cc. The distillate, after the addition of one or two drops of a 1 per cent aqueous solution of phenol, is poured carefully on concentrated sulphuric acid in a large test tube. The presence of even a trace of formaldehyde is indicated by the formation of a crimson color at the plane of union of the two liquids, while, if the preservative be present in greater quantity, a white turbidity, or even a light-colored precipitate, is formed above the red ring.

RECOMMENDATIONS.

The referee would recommend—

(1) That the report be referred to a committee, to be appointed, who shall examine it in detail, paying special attention to the standards which are given, and report in time to have the methods printed in the forthcoming bulletin.

(2) That the referees for the ensuing year pay some attention to the determination of starch in spices. Where they are adulterated by starch products, it has been found possible, even by direct conversion with hydrochloric acid, to detect such

adulterations. It is probable that the use of a modern method would afford a much better criterion.

Also that the referees for next year pay further attention to standards not covered by the referees of this year and to methods for the examination of some substances not taken up by the present referee.

(3) That the title of the referee be changed to "Referee on food adulterants."

THURSDAY—AFTERNOON SESSION.

Called to order by the president at 2.15 p. m.

The chair called for the report of the committee on abstracts, which were read by the chairman of the committee, Mr. Allen.

REPORT OF COMMITTEE ON ABSTRACTS.

Mr. President and Members of the Association.

GENTLEMEN: Your committee has the honor to report that its work during the past year has been conducted on the same general plan as in the previous year. After ascertaining the journals accessible to each member of the committee, an assignment of work was made which was intended to be as nearly equitable as the circumstances would permit. The list of journals for which the different members of the committee have been responsible the past year is as follows:

E. W. Allen: Jour. f. Landw.; Landw. Vers. Stat.; abstract part of Chem. Centbl., Analyst, Chem. Ztg., Jour. Chem. Soc., Ztschr. analyt. Chem., and Ztschr. angew. Chem.; and experiment station publications.

J. T. Anderson: Chem. Ztg.; Jour. Lond. Chem. Soc.

W. D. Bigelow: Bull. Assn. Belge des chim.; Forsch. über Lebensmtl.; Rev. intern. Falsif.; Ztschr. physikal. Chem.

B. H. Hite: Ann. chim. et phys.; Monit. Scientif.; Rev. chim. analyt.

B. W. Kilgore: Analyst; Chem. News; Ztschr. analyt. Chem.

W. H. Krug: Papers on sugar published in the German language.

F. W. Morse: Jour. Amer. Chem. Soc.; Ztschr. anorgan. Chem.; Ztschr. angew Chem.

H. J. Patterson: Amer. Chem. Jour.; Boston Jour. Chem.; Jour. Soc. Chem. Ind.; Science News.

F. W. Woll: Milch Ztg.; Ztschr. Nahr. Untersuch. und Hyg.; Ztschr. physiol. Chem.

The publication of the abstracts began in Experiment Station Record, volume 8, No. 4, and has continued quite regularly through the year up to volume 9, No. 3, which is now in press. The printed work of the committee has occupied about 70 pages. Besides the articles which have been abstracted at considerable length, a large number of papers have been noted briefly or by title merely. In most lines it is believed that the field has been covered in a general way and the work is well up to date.

As in the previous year, the work of the abstract committee has been supplemented by Mr. W. H. Beal, of the Office of Experiment Stations, to whom acknowledgment is here made.

E. W. ALLEN,
J. T. ANDERSON,
W. D. BIGELOW,
B. H. HITE,
B. W. KILGORE,
W. H. KRUG,
F. W. MORSE,
H. J. PATTERSON,
F. W. WOLL,
Committee.

Upon motion the report was accepted.

The chair called for the report of the committee on volumetric standards, which was read by Mr. Kilgore.

REPORT OF COMMITTEE ON STANDARDS FOR VOLUMETRIC APPARATUS, STANDARD TEMPERATURES FOR SPECIFIC-GRAVITY DETERMINATIONS, ETC.

In view of the numerous methods in use by the manufacturers of chemical glassware, for the calibration and graduation of volumetric apparatus, it seems proper that this association, in collaboration with the other scientific bodies of this and other countries, take up the work of bringing about uniformity in the apparatus offered for sale by dealers and in obtaining legislation, if necessary, to insure the marking of all measuring vessels and instruments in a manner that will clearly indicate the system of graduation used, in the case of each individual piece of apparatus.

This subject has been carefully studied in Europe for several years, and several reports have been made by German chemical societies and by committees of the international congresses of applied chemistry which have been held in Brussels and Paris during recent years. The imperial bureau of weights and measures of Germany has taken up the matter, and has minutely defined the conditions under which it works, in testing apparatus submitted for that purpose. A scale of fees has already been established for the testing of apparatus and the placing of the official stamp thereon. A committee is to report on this subject to the next Congress of Applied Chemistry, to be held in Vienna next year.

We therefore make the following recommendations:

(1) That this association adopt, at the earliest moment practicable, a definite system for the graduation of all chemical measuring instruments, including accurate definitions of the basis of graduation, form, and limits of accuracy of each class of apparatus.

(2) That an invariable standard unit of volume be adopted. For this standard we recommend the true liter (the volume occupied by 1,000 grams water at 4° C., weighed in vacuum) and the decimal divisions thereof, which have already been adopted by the International Congress of Applied Chemistry held in Paris in 1896. This is the only invariable and truly scientific unit of volume.

(3) That the members of this association, whenever they make purchases of laboratory supplies in the future, insist upon having apparatus graduated upon this system; also that members of the association having college duties use this system, whenever possible, in their class work.

(4) That the members of this association observe the temperature of their laboratories during working hours, in order to arrive at the best temperature for the calibration of volumetric apparatus for use in their own laboratories and for general use in this country.

(5) That a committee be appointed to take steps to secure the cooperation of other scientific bodies in this work.

(6) That there be printed in the proceedings of this association translations of the requirements for measuring instruments adopted by the Deutsche Gesellschaft für angewandte Chemie in 1891 and published by the German bureau of weights and measures in 1893.[1]

(7) That the results of all determinations of specific gravity, whenever practicable, be expressed in terms of water at 4° C.—that is, that the specific gravity of the substance be regarded as the weight in grams of 1 true cubic centimeter of the substance, at the temperature at which the determination is made.

B. W. Kilgore, *Chairman*.
C. L. Penny.
E. E. Ewell.

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[1] For this translation, see Appendix.

Mr. Patrick moved that the report be accepted and adopted.

Mr. KILGORE. To some members of the association who have not thought a great deal about it, this may not seem an important matter. But when we consider the two systems of volumetric units upon which our glassware is graduated and the differences that may arise when we make up solutions in apparatus graduated on one system, and use them in burettes graduated on the other, it is a matter of the greatest importance. There are two systems in use, the Mohr liter and the true liter. If part of our apparatus be graduated upon the Mohr system and part upon the true liter we may have trouble. It appears to the committee that the true liter is the only invariable standard, and that it would be well for this association, in conjunction with the other scientific bodies of the country, to recognize this as the true volume and insist upon having all our apparatus graduated upon this system. The main advantage claimed for the Mohr system is that at 17.5° a liter flask is placed upon the balance and 1,000 grams of water weighed without making any correction. It is, however, a very simple matter to make the calculation for a correction that would make it the truth instead of the estimation it is.

One other point we desire to call attention to is the temperature to be used in marking this volumetric apparatus. Now we have apparatus marked on the Mohr system at 15° C., though his first temperature was 17.5° C. There is quite a little difference in a liter flask graduated for water at 15° and 17.5° C.; there is an expansion of about 0.06 cc. It is possible, therefore, for large errors to creep into the work on this account. We think it is possible for the members of this association, by observing the temperatures of their laboratories, to come to a uniform standard of temperature at which we can have all our apparatus marked. Seventeen degrees is too low, and 21° or 22° C. would come nearer working conditions. By a long system of experiments, I have found, in our laboratory, that the average temperature for the working time of the day is 21.8° C. We do not think it wise to take any action in this matter now, but leave it for observation in the different laboratories, and if we can come to some understanding as to the temperature at which we can have our apparatus graduated, it will be a great advantage.

The Chair called attention to the fact that the pending motion was for the acceptance and adoption of the report, and questioned whether the word "approved" should not be used instead. The matter, he believed, would come up before the International Congress of Chemists at Vienna shortly, and if this were so it might be well for the association, without taking final action, simply to approve the general recommendation, leaving the adoption to wait upon the action of the international congress, but continuing the committee or appointing

another to see that the general recommendation of the association be registered with the Congress of Chemists at Vienna.

Mr. KILGORE. That was the object of the committee, not to ask for any definite action from the association in recognizing any standard, but merely to bring the matter before it in a way that would help to bring about an international standard. All we ask is that the report be approved and accepted and the matter go over.

Mr. Patrick thereupon withdrew his motion and moved simply that the report of the committee be accepted. Carried.

Mr. Winton moved that the committee on volumetric standards be continued for another year, since its work could not be completed before the European committees reported, and before the association could take final action on the matter it would be necessary to have the committee report again to the association. He also moved that the committee be enlarged from three to five. Seconded and adopted.

The chair later appointed Messrs. Wiley, of Washington, and Caldwell, of Cornell, the additional members of this committee.

RECOMMENDATIONS AS TO DAIRY PRODUCTS.

Mr. BARTLETT. The first four recommendations of the referee on dairy products are approved by the committee. They are as follows:

The committee recommends—

(1) That the provisional method for determining casein in cow's milk be made official, inserting in line 3 (Circular No. 2, Department of Agriculture), after "add," the following words: "One part of formaldehyde to 2,500 parts of milk," and in the first line of the second paragraph substitute the word "preservatives" for "mercuric chlorid."

(2) That the modified provisional method for determining albumin in cow's milk be substituted as a provisional method for the present provisional method, and that further cooperative work be done on the determination of albumin.

(3) That the magnesium sulphate method for determining casein in cow's milk be made an official optional method.

(4) That under milk (*a*) (Bulletin No. 46) the phraseology of the first and second sentences be changed to read as follows: "Heat to constant weight from 1 to 2 grams of milk in a tared flat dish, having a diameter of not less than 5 centimeters, containing 15 to 20 grams of pure dry sand, or without sand, at the temperature of boiling water."

DISCUSSION OF FOOD ADULTERATION.

Mr. BARTLETT. The committee recommends that the report of the referee on food adulterations and standards of purity and the whole subject of food adulterations, including dairy products, be referred to a committee of five, to be appointed by the incoming president, such committee to have subcommittees, and to refer methods and standards to the next meeting of the association.

Mr. BIGELOW. I think probably we might make a little better time than that and get some of the methods into the next bulletin. I think a committee of five would be rather cumbersome; probably a committee

of three would act with more facility. I would move to amend the recommendation of the committee and substitute for it that a committee of three be appointed by the chair to examine the methods and standards reported, modify them as may seem best, and have the report printed in the coming edition of methods of analysis.

Mr. SCOVELL. It was not the intention of the committee to exclude this referee's report from the publication, but we did not wish to adopt any system until this committee reported it. It was the intention to have a committee appointed which would be broad in its scope and have the whole subject of food, especially as to standards, submitted to the next convention. The report of the referee will be included in the proceedings.

Mr. VAN SLYKE. In the adoption of standards for the purity of food stuffs, I think we ought to proceed with caution. If the association adopt standards they will be made use of in the courts of the different States. If we make a mistake we shall do an injury, because our work will be referred to by one side or the other, and if in the suggestion of a standard we make an error, then somebody will make use of it to our injury as well as to the injury of the trade in which that particular product may be found. My feeling is that in the end the best interests of all would be subserved by letting the matter simmer in the hands of a desirable committee for another year.

Mr. HUSTON. There seems to be a little misunderstanding here. I understand that the referee on this subject keeps the same relative position that any other referee does. He deals with the technical work relating to the subject of foods and food adulterants, methods of detection, etc. Now, as I understand it, this committee is to cover more general matters relating to the subject, including whatever action may be necessary relative to legislation, and possibly the passing upon standards for comparison. It seems to me there is a good field for such a committee, and that if drawn from different sections of the country it can be very valuable in this matter, for there are widely varying conditions in even raw material that enters into food products in different sections of the country. I do not understand that this committee is to take the place of the referee at all. I think we should proceed with caution, and that a good, strong, conservative committee can be of service.

Mr. WHEELER. I think we have had points enough to show that this association should proceed slowly in the matter, and time be given for the adoption of standards to which we can adhere in the future. For that reason I hope the amendment will be defeated.

The amendment was lost. The Chair then put the original motion on the adoption of the recommendation, which was carried.

The president-elect, Mr. Winton, appointed the following gentlemen as members of the committee, viz: H. W. Wiley, chairman; H. A. Weber, Columbus, Ohio; M. A. Scovell, Lexington, Ky.; E. H. Jenkins, New Haven, Conn.; William Frear, State College, Pa.

RECOMMENDATIONS ON PURE FOOD AND SOIL ANALYSIS.

Mr. BARTLETT. (Referring to the committee on pure-food legislation.) The committee recommends that this association approve the recommendations of the committee on pure-food legislation, approve the bill as drawn by the committee, that the committee be continued and authorized to transmit the bill to the proper committees of the National House of Representatives and Senate with the indorsement of this association, and urge speedy action along the lines mentioned.

Adopted, and the committee continued with powers as described.

Mr. BARTLETT. (Referring to the recommendations of the referee on soil analysis.) The committee recommends that his first recommendation, that the ammonium and calcium chlorid methods be further investigated next year, be approved.

Adopted.

Mr. BARTLETT. (Referring to the recommendation of the referee on soil analysis, that the one-fifth normal HCl solution for determination of available phosphoric acid be made an official method.) This committee does not recommend the adoption of this as an official method, but that the referee for next year give it further trial.

Adopted.

Mr. BARTLETT. (In regard to recommendations 3, 4, and 5, of the referee on soil analysis.) The committee, after a careful consideration of the referee's paper and examination of the methods printed in Bulletin 49, recommends that the chair appoint a committee to carefully rewrite the methods of soil analysis for the next issue of the official bulletin, the committee to act in conjunction with the secretary.

Adopted. Committee to be nominated by the chair later.

The committee on recommendations withdrew at this moment. The regular order of business was resumed, and papers and communications upon adulteration of dairy products were called for. None was presented.

Papers and oral communications on fermented and distilled liquors were called for. None was presented.

Papers and oral communications on the general subject of food adulterations, and upon analysis of normal dairy products were called for. None was presented, but the following statement was made by Mr. Patrick:

Mr. PATRICK. I have recently discovered that the official methods do not contain the Wollny method for the saponification of fats. It seems to me it should be included. It is saponification under a reflux condenser. As I think it must have been omitted by mistake, I move that it be included in our methods as a provisional method for the coming year, the recommendation to be referred to the committee on recommendations.

Seconded.

Mr. LINDSEY. Can anyone tell us why it was omitted?

Mr. WILEY. The present method of operation was suggested and used on account of its greater rapidity and the absolute certainty of no loss of volatile acids during saponification. I think any of you who have worked with the condenser will have noticed the odor of butyric acid in the room during the operation. Any process which would stop that entirely was thought to be better. Again, saponification under pressure takes less time, and in other respects was found to be more desirable. I should have no objection, however, to allowing persons who wish to use the older method being allowed to do so as a provisional method.

Mr. PATRICK. While I have no fault to find with the pressure method, I believe that where one chemist uses the pressure method half a dozen use the reflux condenser. Since I have been at this meeting I have come across two gentlemen who use the reflux condenser. I myself always used it until within the last year. I should like to hear an expression from other members.

The PRESIDENT. Has anyone made a specific comparison of the two methods? If not, it is a study worthy of attention at any rate.

Mr. WILEY. I think for most fats the reflux condenser is more simple, because a special flask is required for the pressure method. As Mr. Patrick says, for ordinary work perhaps the reflux condenser is more convenient, and for fats without volatile acids is perfectly safe. I do not remember now whether we ever made any comparisons at that time or not.

Adopted, for reference to the committee on recommendations.

No communications were presented upon the subject of fermented and distilled liquors.

The PRESIDENT. Are there any communications upon the subject of analysis of food products? It will be recalled that there is no report from the referee on analysis of sugar. Are there any papers to present upon that subject?

Mr. WILEY. I think the methods are very satisfactory and can be safely left without change. I will say here that the Treasury Department has lately gone very carefully over the methods of polarization, in connection with the tariff on sugar, and there is now in press a pamphlet giving the conclusions of the committee appointed for this purpose by the Secretary of the Treasury, consisting of myself as chairman, Mr. Braid, of the office of weights and measures of the Coast and Geodetic Survey, and Mr. Crampton of the Internal Revenue. We have gone very carefully over the methods of polarization, and while there have been a few minor changes, they are not of sufficient importance to warrant any changes at this time. The Treasury has authorized the use of the shadow instrument, to the exclusion of the old color instrument, which was unsuited to persons who are at all color-blind. Anyone who can see can distinguish a shadow from an illumination, and that is the chief advantage the shadow instrument has over the

color instrument. I believe the members of the association almost universally use the shadow instrument, but those who are not color-blind and use the color instrument doubtless can secure just as accurate results in polarization. Some improvements have been made, and I would advise any of you who are purchasing instruments now to get the triple-field shadow instrument as distinguished from the old double shadow instrument.

An important thing is that any person, connected with official work in the United States, can have his apparatus for polarization standardized by the office of weights and measures of the Coast and Geodetic Survey, free of charge. I advise members of this association to correspond with that office, and as they have opportunity have all their apparatus standardized at that place.

Mr. Huston moved that the Secretary of the Treasury be requested to forward copies of his bulletin on polarization to all members of this association. Seconded.

Mr. WILEY. I think the Secretary of the Treasury will be glad to do so. If this resolution pass, I will myself ask him to grant this favor, and am quite sure he will be glad to do so.

The resolution was adopted.

The PRESIDENT. In the absence of the committee on recommendations, the next subject is the report of the referee on tanning materials. The referee is unable to be present, but the associate referee will present the report.

Mr. Yocum read the following report:

REPORT ON TANNINS.

By JOHN H. YOCUM, *Associate Referee.*

Mr. Westenfelder, the referee for this year, sent out samples of tanning extract to various members of the association. These samples were accompanied by hide powder and a circular letter outlining the method to be pursued. Unfortunately, Mr. Westenfelder found he could not attend the meeting and turned the matter over to me as assistant referee to make the report, etc. Results were returned by five operators and are of a very satisfactory nature. Owing to a misunderstanding, last year's referee did not present a report. I made request to him for the data then collected that they might be embodied in this report, but have not received them.

The method outlined in the circular letter is as follows:

(1) Weigh about 20 grams of the sample; wash into flask with hot water, add cold water nearly to mark, cool, and make up to 1,000 cc; shake thoroughly.

(2) Immediately measure out 100 cc into a weighed dish and evaporate and dry to constant weight at temperature of boiling water. Result: Total solids.

(3) Filter about 125 cc of solution through a doubled folded filter (S & S No. 590, 25 cm), returning filtrate through filter twice. Evaporate and dry as before. Result: Soluble solids.

(4) Shake 20 grams hide powder with 250 cc of water for five minutes; strain through linen. Repeat operation three times. Finally remove as much water as possible by squeezing the material in a press. Transfer the pressed hide powder to a covered dish and weigh. Weigh off a portion approximately equal to one-fourth

of the whole and dry to constant weight. To 200 cc of original solution, add the remainder of the washed hide powder, and shake for ten minutes; add 5 grams of $BaSO_4$ free from soluble salts and shake for one minute; filter immediately through a folded filter (S & S No. 590, 15 cm), returning the first 25 cc. Filtrate must be absolutely clear; if not, repeat filtration till so. Evaporate 100 cc and dry as before. The weight of residue must be corrected for the dilution caused by the water contained in the hide powder. The shaking must be done in some form of mechanical shaker. The machine used by druggists and known as the "milk shaker" is recommended. Result: Nontannins.

(5) Shake 10 grams of hide powder with 200 cc of water for five minutes; filter through linen; squeeze out thoroughly by hand; collect hide powder and repeat operation twice with the same quantity of water. Pass the last filtrate through paper until a perfectly clear liquid is obtained. Evaporate 100 cc and dry as before. Residue should not amount to more than 10 mg. The above is test for quality of the hide powder.

(6) Test a small portion of the nontannin filtrate with a few drops of a 10 per cent solution of gelatin (Nelson's). Solution must remain unaltered (not the slightest turbidity).

(7) Test for soluble hide: Use a filtered solution (original) of the tanning material instead of the gelatin solution. There must be no turbidity.

(6*a*) If the above test show the presence of tannin, repeat whole operation with 25 grams of hide powder.

(7*a*) If above test show the presence of soluble hide, repeat whole operation with a perfectly washed hide powder.

All dryings must be done at the temperature of boiling water, viz, 97° to 99° C.

Dishes should be covered during drying and cooled in desiccators.

One hundred cubic centimeters of solution should be taken in all cases for evaporating and drying.

Dry 5 grams of sample in flat-bottom dish to constant weight for moisture.

The results reported are shown in the table. The maximum difference in total solids is 1.63 per cent; in soluble solids, 1.80 per cent; in nontannins, 0.95 per cent.

Heretofore the major differences have been in the nontannins, and the concordance in this particular in these results is very encouraging.

The soluble hide in the hide powder is reported as follows:

	Gram.
Westenfelder	0.0050
Carr	.0030
Haley	.0010
Krug	.0088
Yocum	.0030

Table of results.

Operator.	Water.	Total solids.	Soluble solids.	Non-tannins.	Tannins.	Reds.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Westenfelder	57.74	42.26	41.02	16.52	24.50	1.24
Carr	59.21	40.83	39.22	16.96	22.76	1.61
Haley	58.69	41.31	40.63	16.68	23.95	.68
Krug	58.99	41.01	40.32	16.95	23.37	.69
Yocum	58.21	42.15	39.86	17.47	22.39	2.34
Mean	58.57	41.51	40.21	16.92	23.39	1.30
Maximum difference from mean	.83	.75	.99	.55	1.21	1.04

Mr. Westenfelder reports that 10 grams of hide powder gave less than 5 mg of soluble material under conditions of testing hide. He obtained 19.18 per cent non-

tannins by shaking 200 cc of the extract solution with 10 grams of dry unwashed hide powder.

Mr. Carr made report of 19.18 per cent of nontannins originally, but later found upon going over his results an error in them, and reports 16.96 per cent nontannins as the correct result.

He dried the materials in vacuo at 100° C., which no doubt makes his results on solids a trifle lower than the others.

Mr. Haley obtained 15.75 per cent of nontannins with the use of 50 grams wet-pressed official hide powder (equivalent to 10½ grams dry); 15.85 per cent with 50 grams of wet-pressed Vienna hide powder (equivalent to 12 grams dry), and 16.24 per cent with 50 grams wet-pressed hide powder of his own make (equivalent to 11½ grams dry).

Mr. Krug obtained 16.24 per cent nontannins by the use of an unofficial hide powder that gave 0.0065 gram of soluble material.

I obtained 17.76 per cent nontannins from an unofficial hide powder.

These results show that, in the hands of operators accustomed to the work, the nontannins can be obtained in better accord than can the results from simple drying of viscous solutions, such as the extract itself is.

It is generally recognized that the tannin determination is not one of a single substance, but rather of a group having much the same characteristics, and that besides the chemical union of tannin and hide there also occurs an occlusion by the hide of the other soluble materials in the extract. This precludes the possibility of obtaining anything like the exact concordance that is obtained by purely chemical methods.

The nontannins, obtained by the various operators using the official and other hide powders on the official extract, show the variations possible with hide powders, answering their individual requirements, and emphasize the necessity of some standard in this respect.

The differences in the results obtained on water and total solids are due to the same general causes that occasion differing results in the drying of other organic solids, and can be regulated to a great extent by uniformity of time and temperature of drying.

Because of the oxidation and decomposition undergone by organic matter when dried in the air it is impossible to obtain final results that represent the true percentage of water, yet most chemists making tannin determinations have not the facilities for drying in hydrogen or in vacuo, and it is as yet an open question if such treatment would prevent decomposition in tanning solutions; consequently it does not seem proper to suggest changes in this direction.

By specifying the length of time, temperature, and size of dish a better agreement may be obtained next year.

The soluble solids are influenced by the temperature of the solution when filtered. Inasmuch as the temperature of the laboratory seldom varies outside the limits of 16° to 20° C., this has little influence. The filter paper causes differences, but S & S No. 590 15 cm seems to give results of a satisfactory nature.

There seems to be more cause for difference in the temperature of the solution at the time the extract is dissolved than from either of the above causes. A solution made by dissolving 20 grams of extract in 1,000 cc of water at 16° C. stood four hours, and filtered through doubled folded 15 cm S & S No. 590 paper gave 39.09 per cent of soluble solids. The same solution standing eighteen hours gave 39.24 per cent soluble solids. Evidently the reds that are first thrown down by contact with cold water are to a limited extent redissolved on standing. By dissolving the extract at 80° C. and letting stand four hours 40.05 per cent soluble solids were obtained, and by letting stand eighteen hours, 40.07 per cent. By dissolving at 25° C. and letting stand four hours 39.75 per cent soluble solids were obtained, and by letting stand eighteen hours 39.62 per cent. By filtering with kaolin the solution made at 16° C. gave 37.54 per cent, that made at 25° C. gave 37.65 per cent, and that made at 80°

gave 38.55 per cent soluble solids. The nontannins were not influenced by any of the methods of dissolving, nor were they less from the kaolin filtrates.

Though the results upon nontannins from this year's work are in fair accord, the variations due to the use of other than the official hide powder are wide. The lowest result is from Mr. Haley—15.75 per cent—and the highest my own—17.76 per cent.

The value of a standard hide powder or a means of knowing the efficiency of a given hide powder, is great. Six hide powders were tested according to the two tests given in last year's published method (Bull. 49, p. 118), the soluble-hide test and the gallo-tannin test.

Results of the year's work are given in the following tables:

TABLE I.—*Analysis of hide powders.*

Sample.	Moisture.	Fat.	Ash.	Al_2O_3 and Fe_2O_3.	CaO.	MgO.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
No. 1	13.00	.....	0.50	.....	.....	.....
No. 2	12.40	0.49	2.66	0.25	2.61	0.04
No. 3	13.00	.39	.45	.10	.19	.02
No. 4	12.30	1.07	.17	.08	.07	.01
No. 5	14.00	.94	.48	.10	.11	.01
No. 6	12.00	.65	.73	.15	.18	.02

TABLE II.—*Comparison of washing tests on various hide powders.*

Sample.	Quantity of air-dry hide.	100 cc, first washing.	100 cc, second washing.	100 cc, third washing.	100 cc, fourth washing.	Hide removed by four washings.
	Grams.	*Grams.*	*Grams.*	*Grams.*	*Grams.*	*Per cent.*
No. 1	20	0.0634	0.0134	0.0093	0.0076	1.31
No. 2	20	.1719	.0375	.0165	.0105	3.42
No. 3	20	.2192	.0525	.0196	.0096	4.32
No. 4	20	.0720	.0172	.0154	.0049	1.56
No. 5	20	.2256	.0415	.0098	.0058	4.10
No. 6	20	.1172	.0254	.0110	.0073	2.27

TABLE III.—*Comparison of gallotannin test on various hide powders.*

Sample.	Weight of air-dry hide.	Weight of wet hide.	Dry hide in wet hide.	Weight of dry hide.	Dilution correction.	Weight of the nontannins.	Corrected nontannins.	Tannin absorbed.
	Grams.	*Grams.*	*Per ct.*	*Grams.*		*Grams.*	*Per ct.*	*Per ct.*
No. 1	20	27.70	44.40	12.30	1.077	0.0069	1.38	98.62
No. 2	20	33.70	27.00	9.10	1.123	.0320	6.42	93.58
No. 3	20	37.10	28.00	10.39	1.134	.0247	4.96	95.04
No. 4	20	32.20	35.00	11.27	1.105	.0120	2.41	97.59
No. 5	20	37.40	31.50	11.78	1.128	.0157	3.13	96.87
No. 6	20	47.30	22.90	10.83	1.182	.0468	9.40	90.60

In Table I is given the analysis of these hide powders, and in Table II the result of washing according to the method; in Table III the results of the gallotannin tests.

Hide No. 2 would be judged poorly prepared from its high ash, and is outside the limits designated by the method for soluble hide. Hide powders Nos. 2 and 6 are outside the limits designated by the gallotannin test.

Hide No. 1 is this year's official hide powder. I did not have enough of it to carry on the full comparison. This left Nos. 3, 4, and 5 as satisfactory hide powders. These powders were used to make comparative determinations on an extract with the following result:

Sample.	Nontannins.
	Per cent.
No. 2	18.82
No. 3	15.70
No. 4	14.72
No. 5	14.45
No. 6	18.58

Nos. 3, 4, and 5, which have satisfied the requirements, give fairly concordant results on the extract.

A further test was made by shaking for ten minutes 5 grams of air-dry hide powder with 200 cc of water, containing 5.48 grams of gallotannin.

Gallotannin per gram of hide powder.

Sample.	Gram.
No. 2, hide powder removed	0.0943
No. 3, hide powder removed	.2393
No. 4, hide powder removed	.3632
No. 5, hide powder removed	.6230
No. 6, hide powder removed	.1448

These hide powders, when completely tanned, do not show any great difference in the amount of tannin absorbed; hence it is a condition in the preparation of the hide powder that gives the more rapid absorptiveness.

These tests when applied to hide powders enable the chemist to keep a fair check on various makes of powder, and assure himself of the uniformity of a given maker's product, but more satisfactory would be the acceptance of some maker's product as a standard.

Your referee would suggest that some minor details of the method be changed and that the hide-powder tests be made to conform with the method relative to washing the hide powder. With this in view, I would recommend (Bul. 49, p. 118) the following as the coming year's method:

RECOMMENDED TANNIN METHOD.

I.—*Preparation of sample.*

Barks, woods, leaves, dry extracts, and similar tanning materials should be ground to such a degree of fineness that they can be thoroughly extracted. Fluid extracts must be heated to 50° C., well shaken, and allowed to cool to room temperature.

II.—*Quantity of material.*

In the case of barks and materials requiring extraction, use such amount of material as will give about 1 gram of solids per 100 cc of solution. In the case of extracts,

weigh 20 grams, dissolve in 900 cc water at 80° C., let stand for twelve hours, and make up extract to 1,000 cc with water.

III.—*Moisture.*

Dry 3 grams of the sample, if it be an extract, in a flat-bottomed dish not less than 2½ inches in diameter to constant weight; if bark, etc., dry 10 grams.

IV.—*Total solids.*

Shake the solution, and without filtering immediately measure out 100 cc with a pipette, evaporate in a weighed dish, and dry to constant weight, at the temperature of boiling water. Dishes should be flat bottomed, and not less than 2½ inches in diameter.

V.—*Soluble solids.*

Filter about 125 cc of the solution through a double-folded filter (S. & S., No. 590, 15 cm), returning all the filtrate through once. To prevent evaporation, funnels should be inserted in small-necked flasks and covered with watch glasses during the filtration. To determine total solids, evaporate 100 cc of filtrate to dryness.

VI.—Nontannins.

Prepare 20 grams of hide powder by shaking for five minutes with 250 cc of water and straining through linen. Squeeze the magma thoroughly by hand, repeat this operation three times, finally remove as much water as possible by means of a press, weigh the pressed hide and take approximately one-fourth of it for moisture determination. Weigh this fourth carefully and dry to constant weight. Weigh the remaining three-fourths carefully and add them to 200 cc of the original solution; shake ten minutes, and squeeze the tanned hide through linen. Collect this filtrate, add 5 grams of kaolin free from soluble salts, stir well and filter through folded filter (S. & S. No. 590, 15 cm), returning the first 25 cc. Evaporate 100 cc of the clear filtrate. The weight of this residue must be corrected for the dilution caused by the water contained in the pressed hide powder. The shaking must be done in some form of mechanical shaker. The simple machine used by druggists, and known as the milk shake, is recommended.

VII.—*Tannins.*

The amount of these is shown by the difference between the soluble solids and the corrected nontannins.

VIII.—*Testing hide powder.*

A. Shake 10 grams of hide powder with 250 cc of water for five minutes, strain through linen, squeeze the magma thoroughly by hand; repeat this operation three times, pass the last filtrate through paper (S and S, No. 590, 15 cm) till clear, evaporate 100 cc, and dry. If this residue amount to more than 10 mg, the hide must be rejected.

B. Prepare a solution of pure gallo-tannin by dissolving 5 grams in 1,000 cc of water. Determine the total solids by evaporating 100 cc of this solution and drying to constant weight. Treat 200 cc of the solution with hide powder exactly as described in paragraph 6. The hide powder must absorb at least 95 per cent of the total solids present. The gallo-tannin used must be completely soluble in water, alcohol, acetone, and acetic ether, and should not contain more than 1 per cent of substances not removed by digesting with excess of yellow mercuric oxid on steam bath for two hours.

IX.—*Testing nontannin filtrate.*

A. For tannin.—Test a small portion of the clear nontannin filtrate with a few drops of a 1 per cent solution of Nelson's gelatin. A cloudiness indicates the presence of tannin, in which case repeat the process described under 6, using 25 instead of 20 grams of hide powder.

B. For soluble hide.—To a small portion of the clear nontannin filtrate add a few drops of the filtered tannin solution. A cloudiness indicates the presence of soluble hide, in which case repeat the process described under 6, giving the hide powder a more thorough washing.

The temperature of solutions when measured or filtered should not be below 16° C. or above 20° C. All drying should be made in flat-bottomed dishes of at least 2½ inches diameter. S and S, No. 590, 15 cm filter paper should be used on all filtrations.

Mr. Forbes, of Arizona, presented the following paper:

PREPARATION OF TANNING EXTRACTS FOR ANALYSIS.

By Robert H. Forbes.

While perfecting a method for the determination of tanning materials in canaigre, the writer was led to make some observations on the method of extraction, which are of general application.

Much of the inaccuracy of the hide-powder method, as applied to barks, woods, leaves, etc., is without doubt due to the manner of extraction laid down in the official methods. It is there advised that the ground material be digested over night at 80° with 500 cc of water. Under these circumstances a loss of tanning materials takes place, which is due to their slow recombination with the cellulose, starch (if present), and albuminoids of the material under examination.

Since the time of digestion, being "overnight," is not constant, and since the condition and amount of cellulose, starch, and albuminoids are variable, the amount of error due to their slow recombination is very irregular. In order to avoid this recombination, it is evidently necessary to devise a method by which the dissolved tannins shall be removed as promptly as possible from the extracted residue.

Extraction by percolation is a simple and nearly perfect way to accomplish this, and it is noticed on page 118 of the Proceedings of the Association for 1896 that it is recommended by the committee to extract barks, woods, leaves, etc., in a Sohxlet or similar apparatus, no details, however, as to temperature and time of extraction being given.

These considerations are of importance, inasmuch as certain materials containing difficultly soluble matters, such as hemlock bark, can not be completely extracted at lower temperatures, while canaigre, which contains starch, can not be percolated at above 50° to 55° C., because of the solution of starch with its consequent inaccuracies.

The following work was carried through with oak, hemlock, and canaigre in order: (1) To establish the fact of error due to recombination of tannings with exhausted residues when these are left in contact with the water extract. (2) To test the percolation method of extraction and note the modifications of temperature needful with different materials.

The reabsorption of tanning materials was established in the following way: A Sohxlet extractor was fitted with a 100-mesh brass wire gauze disk pushed nearly to the bottom of the tube, and a mat of woolly asbestus (previously extracted with HCl) deposited on the gauze and covered with fine asbestus.

The extractor was then placed in a sheet-metal tank of suitable dimensions and 8 to 10 inches deep, the bottom being perforated with holes which receive the rubber stoppers, through which the lower ends of the Sohxlets project. The tank was then filled with water of the desired temperature and kept at that temperature by a flame below. A weighed amount of finely ground material, 5 to 10 to 20 grams, was

floated into the extractor, which was then fitted at the top with a tightly fitting rubber stopper carrying a 250 to 500 cc drop funnel. The funnel was filled with water and the rapidity of the percolation controlled by the stopcock.

The percolates (500 cc) were divided into two equal portions. One portion was examined as it stood for tannins and nontannins, while to the other, half of the extracted residue (a proportionate part) was added, the whole was digested overnight at 80° in a closed flask, the liquor cooled, filtered, and tannins and nontannins determined as before. The results were corrected for dilution by the water added in the wet residue. For comparative results, a parallel analysis was made of extracts by digesting the substance overnight at 80° according to the official method. The results were as follows:

Percentages of extracts.

Samples.	In original percolate.	In percolate redigested with extracted residue.	Difference due to absorption of tannins.	In extract obtained by official method.	Advantage of percolation over official method.
Twenty grams oak bark percolated to 500 cc in four hours at 80° C.:					
Tannins	10.99	9.98	1.01	9.68	1.31
Nontannins	5.10	5.30	.....	4.78	.....
Ten grams hemlock bark percolated to 500 cc in five hours at 80° C.:					
Tannins	13.74	11.80	1.94	11.12	2.62
Nontannins	3.94	4.63	.....	4.13	.....
Five grams of canaigre percolated to 500 cc in eight hours at 50° C.:					
Tannins	41.27	[1] 25.02	16.25	34.70	[2] 6.57
Nontannins	13.03	13.66	.....	12.86	.....

[1] This canaigre residue was digested with its liquor at room temperature.

[2] In this case the ground material was in contact with the liquor only two to three hours, at room temperature.

The results in columns 1 and 2 all show a reabsorption of tanning materials by the extracted residues, being greatest in the case of canaigre. The enormous loss with this material may be partly due to the presence of starch, which also reprecipitates tanning materials. The reprecipitation noted in columns 1, 2, and 3 explains the advantage of percolation over the official digestion method of extraction, as shown in columns 4 and 5.

The action of starch and cellulose, respectively, in reprecipitating the tanning materials was observed by preparing a canaigre percolate, dividing into three portions, analyzing portion 1 as obtained, redigesting portion 2 in the cold with 1 gram of shredded filter paper, and redigesting portion 3 in the cold with 1 gram of bruised starch. Portions 2 and 3 were filtered, and in both cases a loss of tannins was observed.

Percentages obtained.

Samples.	Tanning materials in original percolate.	After digestion with cellulose.	Reabsorption by cellulose.	After digestion with starch.	Reabsorption by starch.
No. 1510, old, red canaigre, percolated at 50° C.:					
Tannins	40.58	36.46	4.12	38.64	1.94
Nontannins	12.72	13.04	.....	13.90	.....
No. 1511, young, yellow canaigre, percolated at 50° C.:					
Tannins	31.98	28.50	3.48	30.24	1.74
Nontannins	16.12	15.26	.....	16.52	.....

The cellulose shows a much stronger action than starch, the reprecipitation being about twice as great.

This suggests the inaccuracies introduced into analytical work by the use of filter papers. Our repeated experiments show that the first portions of tanning extracts filtered through paper contain noticeably less solids than the latter portions—the deficiency being found due to loss of tanning materials. In order to do away with this error I now use an asbestus filter made by placing a mat of prepared woolly asbestus in a porcelain strainer funnel, covering the mat with fine asbestus, and drying the filter in an oven. By the use of this filter no loss of tanning materials occurs and the amount of total solids obtained is usually greater than in the last portions of the same liquors filtered through paper.

The effect of temperature upon completeness of extraction was shown by percolating oak, hemlock, and canaigre at different temperatures shown by the table.

Percentages obtained.

Samples.	Percolated at 55° C.	Percolated at 80° C.	Percolated at steam heat.
Oak bark:			
Tannins	9.01	10.99	10.69
Nontannins	4.44	5.10	6.75
Hemlock bark:			
Tannins	10.73	13.74	15.69
Nontannins	3.58	3.94	8.22
Canaigre, old roots:			
Tannins	41.27	.....	41.74
Nontannins	13.03	.....	17.84
Canaigre, young roots:			
Tannins	31.67	.....	30.68
Nontannins	16.25	.....	28.56

It is seen that the extraction of oak bark is complete at 80°, while hemlock, owing probably to the difficultly soluble reds, yields more extractives by continuous percolation at the higher temperature. The extraction of canaigre is complete at 50° to 55° C. It is, in fact, not possible to make a satisfactory percolation of this material above this point, since the starch then swells and clogs up the Sohxlet tube.

The percolations of canaigre at steam heat, noted in the table, were made by placing the material in a thin layer on an asbestus mat in a large continuous copper extractor. The increase in 1510 of nontannins from 13.03 per cent to 17.84 per cent, and in 1511 from 16.25 per cent to 28.56 per cent is due to the solution of starch, whose presence in the extract causes turbid filtrates and is therefore an element of inaccuracy.

The above observations show—

(1) That in the extraction of barks, woods, leaves, etc., a percolation method is essential to complete extraction, owing to the reabsorption of tanning materials by cellulose, starch, albuminoids, etc.

(2) That the percolation method must be modified to suit the material under examination. Continuous extraction at steam heat will probably manage most woody materials, but canaigre requires to be percolated at 50° to 55° C., at which point extraction is complete, and we avoid the solution of starch, which does not properly belong with soluble nontanning materials and whose presence is a source of inaccuracy in determining tannins.

(3) In accurate work the use of an asbestus filter is necessary, the loss of tanning materials in the filter thus being avoided.

(4) The following method of extraction for canaigre is recommended: A Sohxlet extractor is fitted inside with a 100-mesh brass gauze disk which is pushed nearly to the bottom of the tube, and a mat of woolly asbestus, previously extracted with hot hydrochloric acid, is deposited on the gauze. The mat is then covered with fine asbestus, the prepared extractor is placed in a sheet-metal tank of suitable size and 8 to 10 inches deep, the bottom of which is perforated with holes which receive the rubber stoppers through which the lower ends of the Sohxlets project. The tank is then filled with water at 50° C., and kept at that point by a small flame. The extractor is then partly filled with water and 5 grams of canaigre are floated into the extractor. A tightly fitting rubber stopper carrying a 250 to 500 cc funnel is then fitted to the top of the Sohxlet, the funnel is filled with water and the stopcock turned so that the percolate will run to 500 cc in four hours. The percolate is allowed to cool, made to the mark, shaken, an aliquot taken for total solids, the remainder filtered through an asbestus filter and soluble solids and tanning materials determined as usual.

(5) In conclusion, it is advisable that a more detailed examination of the percolation method, as applied to different materials, be made, and the best temperature for each one determined, as it is certain that any particular temperature can not be fixed upon for all substances alike.

Mr. KRUG. As the hour is late, I move that the report be simply given to the committee on recommendations with power to act.

Adopted, and the report so referred.

RECOMMENDATIONS.

The committee on recommendations returned to the hall and made the following additional recommendations:

We recommend the following addition to the provisional method under Dairy Products:

The Wollny method.—Use same amounts of alkali and alcohol as in previous method (pressure method) and conduct the saponification on a steam or water bath, the flask being connected with a reflux condenser not less than one meter in length and kept well cooled during the operation. Saponification will usually be completed within one-half hour.

Adopted.

Mr. Patrick moved that the referee for next year be instructed to test this method. Adopted.

Mr. BARTLETT. The committee recommends the adoption of Recommendation No. 1 of the referee on cattle foods, with the exception that the time of acid treatment be made two and a half hours instead of two hours.

The committee further recommends that the referee for next year test the following procedures—"after the starch reaction has disappeared, cool to 55° and repeat the treatment with diastase as described for thirty minutes."

Adopted, as a provisional method.

Mr. BARTLETT. The committee recommends the adoption of the phloroglucin process as a provisional method for the estimation of furfurol in agricultural products.

Adopted.

Mr. BARTLETT. The committee recommends that in the estimation of

galactan the method described on page 98 of the referee's paper for 1897 be adopted as a provisional method.

Adopted.

Mr. BARTLETT. On page 62, Bulletin 46, under "c," Official Methods, first line, that "two and a half hours" be substituted for "three hours."

Adopted.

Mr. BARTLETT. The committee further recommends the following rearrangement of the provisional methods for cattle foods:

That the provisional methods on page 62 of the Official Methods be rewritten in the following order:

A and B, same as now printed.
C, determination of starch in commercial starches.
D, determination of starch in all other substances, diastase method.
E, determination of pentosans by the phloroglucin method.
F, determination of galactin by method described in 1897, referee's paper, page 25.
G, determination of crude fiber.

Adopted.

Mr. BARTLETT. The committee recommends that the names referee and associate referee be substituted for reporter and associate reporter.

Adopted.

Mr. BARTLETT. The committee recommends that the name reporter on fermented and distilled liquors be changed to referee on liquors and food adulterations.

Adopted.

Mr. BARTLETT. The committee recommends that the executive committee, in addition to fixing the time and place of meeting, be authorized to fix the order of business and see that it is announced in the call for the meeting.

Adopted.

Mr. BARTLETT. The committee recommends that upon the republication of the methods they be all properly named, defined, and arranged in groups in accordance therewith.

Mr. WHEELER. I was the originator of that. In view of the fact that in some States they provide that our official methods shall hold in court, it seemed desirable that our methods should be appropriately grouped together, the provisional as provisional, the optional as optional, and these names defined, so that all may know what is meant by provisional and optional.

Adopted.

The Chair announced as the committee on restatement of the methods of soil and ash analysis Messrs. Scovell, Peter, and Wiley.

RESOLUTIONS.

The Chair called for the report of the committee on resolutions.

Resolved, That the thanks of this association are due—

1. To the authorities of the Columbian University, and especially to President Whitman and Dean Munroe, for the use of their lecture room for our meetings, and for other courtesies.

2. To the honorable Secretary of Agriculture, for the patronage of his Department in the publication of the annual reports of the association and for the encouragement given the association by his presence and generous expressions of good will.

3. To the Cosmos Club, for its hospitality in extending to the members of the association the privileges of the club.

4. To President Frear, for the able, impartial, and uniformly courteous manner in which he has presided over the deliberations of the association.

5. To Secretary Wiley, for his efficient services and his active and continued interest in the work of the association.

J. T. ANDERSON,
W. A. WITHERS,
H. B. MCDONNELL,
Committee.

Adopted.

Mr. R. H. Atwater addressed the convention briefly on the subject of ammonium sulphate, asking the members of the association, as American citizens, to give this substance its true value.

The incoming president, Mr. Winton, read the list of referees for the coming year.

Some discussion arose as to the time of holding the meetings, some of the Southern members desiring that they be held during the summer vacation. Mr. Persons moved that the association recommend to the executive committee that the meeting be held during the summer vacation months. The motion was duly seconded, but failed of adoption.

The Chair, expressing his gratification at the amount of work accomplished and the consideration shown by the members during the meeting, declared the convention adjourned *sine die.*

OFFICERS, REFEREES, AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR THE YEAR 1898.

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A. L. WINTON, New Haven, Conn.

VICE-PRESIDENT.

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SECRETARY.

H. W. WILEY, Washington, D. C.

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J. L. HILLS, Burlington, Vt.

REFEREES.

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Nitrogen.—Mr. R. J. Davidson, Blacksburg, Va.
Potash.—Mr. C. H. Jones, Burlington, Vt.
Soils and ash.—Mr. Harry Snyder, St. Anthony Park, Minn.
Dairy products.—Mr. C. L. Penny, Newark, Del.
Foods and feeding stuffs.—Mr. W. H. Krug, Washington, D. C.
Liquor and food adulteration.—Mr. W. S. Sweetser, State College, Pa.
Sugar.—Mr. R. S. Hiltner, Lincoln, Nebr.
Tannin.—Mr. John H. Yocum, New York, N. Y.

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Nitrogen.—Mr. F. S. Shiver, Clemson College, S. C.
Potash.—Mr. B. B. Ross, Auburn, Ala.
Soils and ash.—Mr. B. L. Hartwell, Kingston, R. I.
Dairy products.—Mr. J. B. Weems, Ames, Iowa.
Foods and feeding stuffs.—Mr. G. L. Teller, Fayetteville, Ark.
Liquor and food adulteration.—Mr. Chas. P. Worcester, Boston, Mass.
Sugar.—Mr. Elton Fulmer, Pullman, Wash.
Tannin.—Mr. Oma Carr, Corry, Pa.

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Mr. H. W. Wiley, Washington, D. C., chairman.
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Committee on Uniform Laws for Fertilizer Control.

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Mr. B. B. Ross, Auburn, Ala.

Committee on Volumetric Standards.

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Mr. C. L. Penny, Newark, Del.
Mr. E. E. Ewell, Washington, D. C.
Mr. G. C. Caldwell, Ithaca, N. Y.
Mr. H. W. Wiley, Washington, D. C.

Committee on Rewriting Methods of Soil Analysis.

Mr. M. A. Scovell, Lexington, Ky., chairman.
Mr. A. M. Peter, Lexington, Ky.
Mr. H. W. Wiley, Washington, D. C.

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists of the United States. The objects of the association shall be (1) to secure uniformity and accuracy in the methods, results, and modes of statements of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership; and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to enter motions or vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. Any person eligible to membership may become a member at any meeting of the association by presenting proper credentials and signing this constitution. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled to become honorary members, and to have all privileges of membership, save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote.

(3) The officers of the association shall consist of a president, a vice-president, and a secretary, who shall also act as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership, his office shall be considered vacant, and a successor may be appointed by the executive committee, to continue in office till the annual meeting next following.

(4) There shall be appointed by the president, at the regular annual meeting, a referee and associate referee for each of the subjects to be considered by the association.

It shall be the duty of these referees to prepare and distribute samples and standard reagents to members of the association and others desiring the same; to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) No changes shall be made in the methods of fertilizer analyses, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of fertilizer work to test the proposed changes.

(8) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(9) The executive committee will confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(10) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by the approval of two-thirds of the members present entitled to vote.

APPENDIX.

NOTICE IN REGARD TO THE TESTING OF CHEMICAL MEASURING FLASKS, ETC.

[From Zeitschrift für angewandte Chemie, 1893, pp. 557–559. Translated for the use of the committee by E. E. Ewell.]

The Kaiserliche Normal-Aichungs-Kommission published the following directions, based upon article 18 of the regulations governing weights and measures (special supplement to Reich's Gesetzblattes, No. 30):

I. Allowable Measuring Apparatus.

1. For exclusive use for chemical volumetric analysis of aqueous solutions, glass vessels will be admitted for testing as follows:

Vessels with one mark to hold a single volume.

(*a*) Flasks.

(*b*) Pipettes with upper suction tube and a lower delivery tube.

Vessels marked to hold a certain definite volume and certain subdivisions thereof.

(*c*) Measuring glasses (also called measuring cylinders, being measuring tubes having a foot).

(*d*) Burettes (measuring tubes without a foot, but with a delivery tube).

(*e*) Measuring pipettes (measuring tubes having a suction tube at the upper end and a delivery tube at the lower end).

2. The capacity of a measuring vessel is to be limited either by the mark or the lower opening, and its quantity is marked upon the vessel for a temperature of the vessel of 15° of the centigrade thermometer, in the liter, fractions of a liter, or in cubic centimeters, in which case the cubic centimeter is to be the 1–1000 part of a liter.

3. Measuring vessels may be graduated either to hold (*Messgeräthe auf Einguss*) a given quantity to the mark, or to deliver (*Messgeräthe auf Ausguss*) a given quantity. Measuring vessels provided with a device for the delivery of liquids must always be graduated to *deliver* the volume of liquid for which they are marked. Other vessels may be graduated to either hold or to deliver, but for only one of these purposes in the case of any single vessel. The volume of liquid is measured with a delivery apparatus by filling and emptying, with precautions to retain a uniformly unpreventable moistening of the walls of the vessel. This uniform moistening is insured by observing the following precautions:

(*a*) Measuring vessels which are emptied by turning upside down are held for one minute after emptying in an inclined position to allow them to drain, when the last drop is wiped off.

(*b*) In the case of pipettes the liquid is allowed to flow out entirely, or to the lower mark, according to the method of graduation, while the delivery tube is held without motion in contact with the sides of the receiving vessel. After the free

outflow of the liquid has stopped, or after the lower mark has been reached, the pipette is allowed to drain for one-quarter of a minute.

(*c*) In the case of burettes, or measuring pipettes (pipettes graduated to deliver fractions of their maximum capacity), the desired quantity is drawn out, the last drop is removed, and the reading is taken after waiting two minutes.

4. The cross section of the measuring vessel must always be circular. The space occupied by the liquid to be measured, viewed in the direction of its greatest diameter, should decrease in size above and below, best at the same rate. Where the body of the vessel has a tube or cylindrical portion sealed on, the body of the vessel must gradually merge into the cylindrical portion without any abrupt change of direction, so that there will be no hindrance to the ready outflow of the liquid.

5. The mark shall be narrow, and both it and the label shall be plainly etched, ground, or applied in some other permanent manner. It should never be merely painted. The mark should always be upon a cylindrical, regularly formed, transparent part of the vessel. Coloring the mark is permitted.

6. The mark shall pass at least halfway around the vessel and lie in the plane to which the axis of the vessel is perpendicular.

7. In the case of measuring vessels marked for subdivisions of their total capacity, these subdivisions must all be equal.

8. Capacity of a flask may be stated in liters or cubic centimeters (the mark being *liter*, *l.*, or *cc*), while all other vessels may be marked only cubic centimeters—i. e., with the mark *cc.* The mark expressing the capacity of a vessel without subdivisions is placed on the middle part of the body of the vessel.

9. The numbering of the marks on vessels marked for subdivisions of their total capacity is to be placed at the right end of the lines denoting cubic centimeters as units. These numbers must run consecutively only from either the top down or only from the bottom up.

The "cc" mark is added to the line bearing the largest number, whether it be at the beginning or at the end of the graduation.

10. There must be etched below the mark indicating the capacity, in the case of flasks and other vessels having but one graduation mark, the expression + 15° C., indicating the temperature at which the capacity of the flask is equal to the volume indicated by its label. In the cases of instruments graduated for fractions of their total capacity, this mark (+ 15° C.) is etched at least 15 mm above the graduation. There must also be placed at the left of this mark and at the same height the mark *E.* or *A.*, *Eing.* or *Ausg.*, or *Einguss* or *Ausguss*, as the case may be, to indicate whether the instrument has been graduated to hold or to deliver the volume of liquid indicated by its label.

A manufacturer's number, name, and address, and a trade-mark may be placed opposite the label mentioned above on the other side of the flask. In the case of apparatus graduated for subdivisions the manufacturer's marks are to be placed in long lines at the left of the graduation.

11. In the case of all measuring vessels the reading shall be taken at that point of the wall of the vessel where it is cut by the plane which is perpendicular to the axis of the vessel and at the same time tangent to the meniscus of the liquid at its lowest point.

12. Inflow and outflow tubes, stoppers, etc., must not end in the measuring space of the vessel or extend therein. The limiting of the measuring space directly by cocks is inadmissible. Beyond the part actually occupied by the liquid to be measured the vessel may be provided with cocks, tubes, expansions made of irregular form, etc., ad libitum.

13. In the case of measuring vessels having a delivery tube or point, the latter should be drawn out as thin as stability will permit, and its termination should be smooth and even. It is permissible to constrict the orifice somewhat. In the case of burettes of the Gay-Lussac form, the delivery point should be bent back toward the delivery tube, and should be ground obliquely on the under side.

II. Measuring Vessels Without Minor Graduation.

(Flasks and pipettes graduated to deliver one maximum volume.)

1. Flasks may be made of the following capacities: 2, 1, ½ (0.5), ¼ (0.25), 0.2, 0.1, and 0.05 liter; pipettes, of any desired capacity from 1 to 200 cc.

2. In the case of both forms of apparatus the part bearing the graduation mark must be cylindrical (see foregoing I, 5), of uniform section, and transparent. Moreover, this cylindrical portion must pass over very gradually into the expanded part of the apparatus.

3. In the case of pipettes the suction tubes must be at least 130 mm long, and the delivery tube at least 60 mm, but not more than 300 mm, long.

4. The graduation mark must be at least 70 mm from the upper end of the neck of a flask, and in the case of a pipette at least 100 mm from the upper end and at least 30 mm from the bulb. The mark must extend entirely around the neck of the flask or the suction tube of the pipette.

5. At the point where the graduation mark is placed the internal diameter of the neck of the flask shall not be less than 6 mm, and shall not exceed the following maximum figures given for flasks of each of the sizes admitted for testing:

Capacity of flask.	Maximum internal diameter of neck.
Liters.	*Mm.*
2	25
1	20
½ (0.5)	20
¼ (0.25)	15
0.2	12
0.1	12
0.05	10

In the case of pipettes the suction and delivery tubes must have an internal diameter of not less than ½ mm and not more than 6 mm.

6. The bottom of the flask may be slightly reentrant. The circumference of the bottom of the flask must form a plane to which the neck of the flask is perpendicular. The flask must stand solidly on a horizontal surface.

7. The lower limit of the volume of a pipette may be either the end of the delivery tube or a second mark etched thereon at least 30 mm from the end.

In the case of pipettes without a cock, the delivery orifice must be of such a size that the discharge of the pipette, conformable to I, 3, (*b*), lasts:

12 to 15 seconds when the pipette is of less than 10 cc capacity.

15 to 20 seconds when the capacity of the pipette is of 10 cc or more but less than 50.

20 to 30 seconds when the capacity of the pipette is 50 cc or more but less than 100.

30 to 40 seconds when the capacity of the pipette is 100 cc or more.

In the case of pipettes provided with cocks, the testing must be made for that setting of the cock which requires the following time for emptying the pipette:

13 to 17 seconds when the capacity of the pipette is less than 10 cc.

16 to 20 seconds when the capacity of the pipette is 10 cc or more, but less than 50.

23 to 27 seconds when the capacity is 50 cc or more but less than 100.

33 to 37 seconds when the capacity of the pipette is 100 cc or more.

III. Measuring Vessels with Minor Divisions.

1. The total volume of vessels graduated for subdivisions thereof may have any value between 1 cc and 1 liter, but for cylinders and burettes shall not be less than 5 cc, and for burettes and pipettes not more than 100 cc.

2. The subdivisions permitted are as follows:

When the total capacity of the apparatus is 1 cc and up to and including 2 cc, the smallest subdivision is 0.01 to 0.02 cc.

When the total capacity of the apparatus is more than 2 and up to and including 5 cc, the smallest subdivision is 0.05 to 0.02 cc.

When the total capacity of the apparatus is more than 5 and up to and including 10 cc, the smallest subdivision is 0.05 to 0.1 cc.

When the total capacity of the apparatus is more than 10 and up to and including 50 cc, the smallest subdivision is 0.1 to 0.2 cc.

When the total capacity is more than 50 and up to and including 100 cc, the smallest subdivision is 0.2, 0.5, or 1 cc.

When the total capacity is more than 100 and up to and including 200 cc, the smallest subdivision is 1, 2, or 5 cc.

When the total capacity is more than 200 and up to and including 500 cc, the smallest subdivisision is 5 to 10 cc.

When the total capacity is more than 500 cc, the smallest subdivision is 10 cc.

3. The measuring space may be limited by lines below as well as above. In the case of pipettes, the upper graduations must be at least 100 mm from the upper end of the apparatus and at least 50 mm in other cases. The lower mark of the graduation, in cases where the bottom of the vessel is not the lower limit of the graduation, must be at least 30 mm from the lower end of the apparatus or from the beginning of the contraction of the body thereof.

4. The numbering is done as follows:

a. In case of division into 10, 1, 0.1, or 0.01 cc, every tenth mark is numbered.

b. In case of division into 2, 0.2 or 0.02 cc, every fifth line is marked.

c. In the case of division into 5, 0.5 or 0.05 cc, every second or tenth line is marked.

The numbered lines must extend entirely around the vessel. Of the other lines, the fifth in the case "a" and whenever only every tenth line is numbered, and the first line when every second line is numbered, must extend three-fifths of the way around the vessel, while all other lines extend only one-half way around.

All lines not passing entirely around the vessel must be on transparent glass; any opaque strips for facilitating the reading of the instrument must not be greater in breadth than two-fifths of the circumference of the tube.

5. The distance between two consecutive division marks must not be more than 12 mm, and for measuring cylinders graduated for each 5 cc or more, not less than 2 mm, while for all other measuring vessels this distance must not be less than 1 mm.

IV. Limit of Error.

1. Measuring vessels without divisions.

For flasks graduated for delivery the error must not exceed the following limits:

Capacity.	Allowable error.
Liters.	*cc.*
2.0	1.0
1.0	.6
.5	.3
.2	.2
.1	.2
.05	.1

For flasks graduated to hold the quantities of liquid just named, the allowable error is one-half as great in each case.

For pipettes delivering only maximum quantity, the allowable errors are as follows:

Capacity of pipette.	Allowable error.
cc.	*cc.*
1 to 2	0.01
More than 2 to 10	.02
More than 10 to 30	.03
More than 30 to 75	.05
More than 75 to 200	.1

2. Measuring vessels with divisions—burettes and pipettes.

Maximum capacity.	Allowable error on maximum capacity.
cc.	*cc.*
1 to 2	0.01
More than 2 to 10	.02
More than 10 to 30	.03
More than 30 to 50	.05
More than 50 to 100	.1

In the case of graduated cylinders of the dimensions just given, the allowable error is double that for burettes and pipettes when the instruments are graduated to hold a certain volume, but four times as great as the errors allowed for burettes and pipettes when graduated for delivery or pouring out.

For larger measuring cylinders graduated to hold definite volumes, the allowable errors are as follows:

Maximum capacity.	Allowable error.
cc.	*cc.*
More than 100 to 200	0.5
More than 200 to 500	1
More than 500	2

When graduated for delivery or pouring, the allowable errors are just twice as great.

For measuring cylinders graduated to hold a definite volume, the maximum errors for the volumes indicated by ten of the consecutive smallest divisions must on no part of the graduation be greater than as shown below:

1 cc when the divisions equal 10 or 5 cc.

0.4 cc when the divisions equal 2 cc.

0.2 cc when the divisions equal 1 and 0.5 cc.

0.1 cc when the divisions equal 0.2 and 0.1 cc.

In the case of cylinders graduated for delivery, double these errors are allowed. In the case of burettes and pipettes graduated to 0.01 to 0.2 cc, the error must not be greater than one-third of the smallest division, and not more than one-fourth of the smallest division in other cases.

V. Stamping.

The official stamp is etched upon flasks immediately over the graduation mark and also over the label. On pipettes graduated to deliver one maximum volume the official stamp is etched immediately over the upper mark and immediately under the lower mark, when there is one.

In the case of other measuring vessels the official stamp is placed close above the upper mark and close below the lower one; besides this, the stamp is also placed on the delivery tube, close to its end.

VI. Fees for Testing.

The fees are as follows:

(*a.*) For testing and placing the official stamp on measuring vessels without divisions, 30 pf. ($0.072).

Measuring vessels having divisions, 80 pf. ($0.192).

(*b.*) For mere testing, for each complete volume or each mark tested, 10 pf. ($0.024.)

If a vessel having divisions is tested for more than 5 marks besides the one indicating the total volume, each additional mark tested is charged for at the rate given under b.

VII. Place of Testing.

The testing and stamping of measuring vessels will be done by the Normal-Aichungs-Commission until further notice.

Huber,
Kaiserliche Normal-Aichungs-Kommission.

Berlin, *July 26, 1893.*

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